Pressure Swing Absorption Device and Process for Separating CO_2 from Shifted Syngas and its Capture for Subsequent Storage

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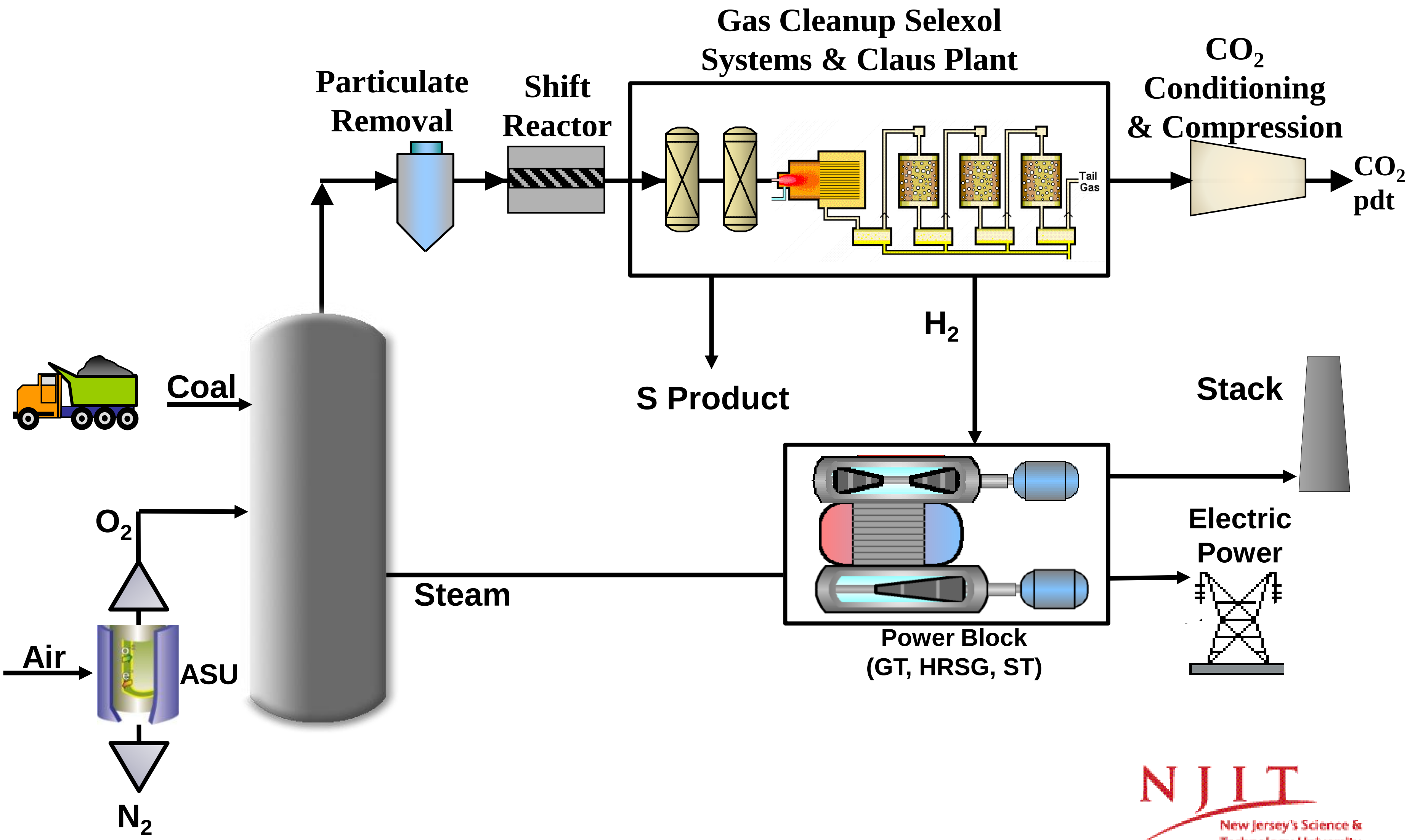
Typical IGCC Plant with a Shift/Selexol based CO_2 Capture System



- **Background**
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Precombustion CO_2 Capture from Shifted Syngas

1. Typical IGCC plant will employ Selexol-based CO_2 capture:
    - Shifted syngas is cooled, humidified and expanded to recover some energy
    - Double-stage Selexol unit will remove H_2S and CO_2
    - H_2S recovered as elemental sulfur in a Claus unit
    - CO_2 flashed off the absorbent liquid at a lower pressure (~50 psia)

2. Variety of Sorbent-based Process, Ca-based, Hydrotalcite-based etc., Thermal swing sorption-enhanced reaction (TSSER), PSA

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3. Membrane Processes:

- Highly H_2-selective metallic or ceramic or zeolitic membranes
- CO_2-selective polymeric membranes
- Membrane reactor for shift gas reaction with CO_2-selective membrane
- Ionic liquid-based supported liquid membranes

4. Pressure swing absorption of CO_2 for post low-temperature shift reactor gas using membrane contactors containing ionic liquids, dendrimers etc.

5. Other processes…

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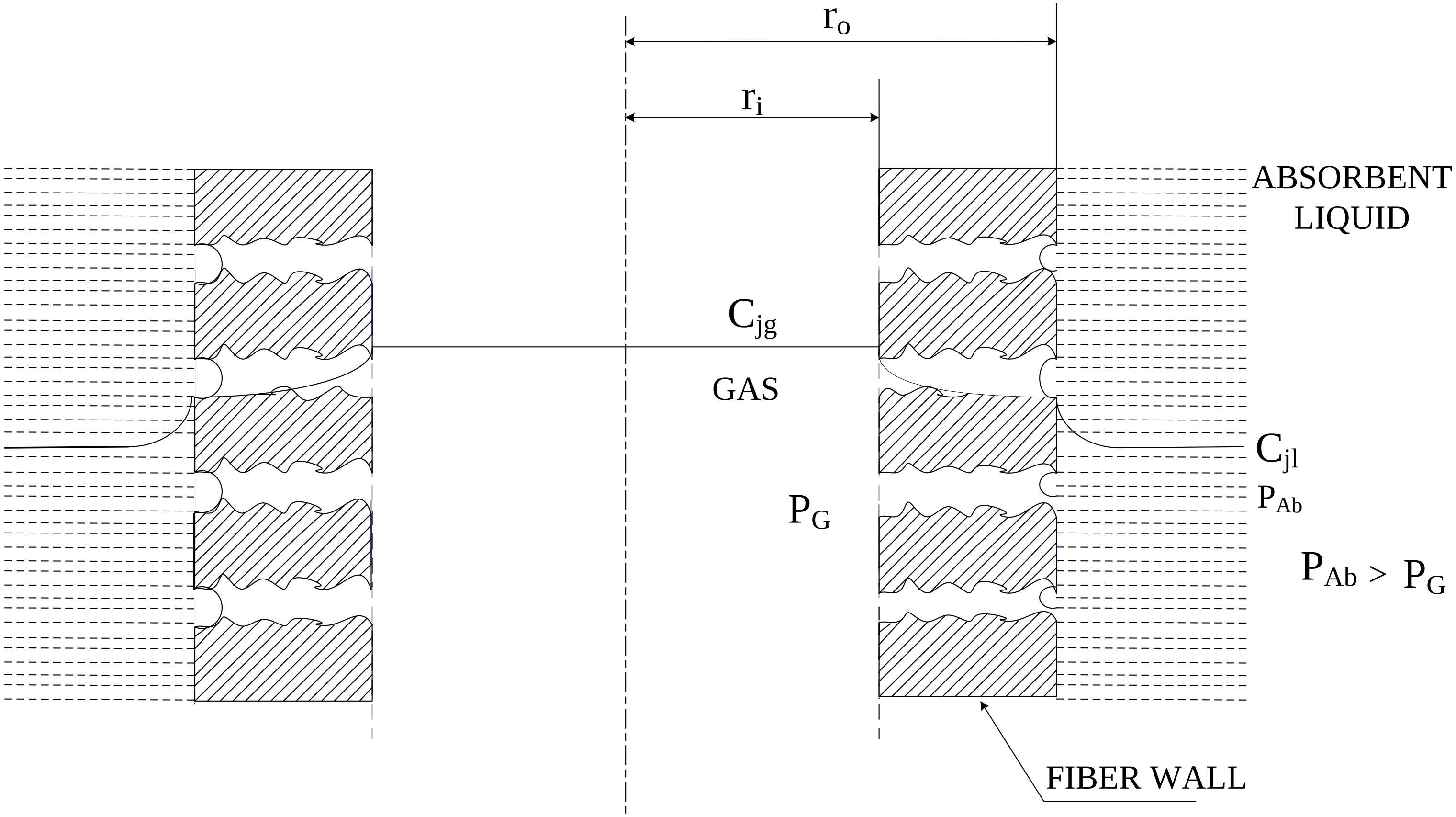
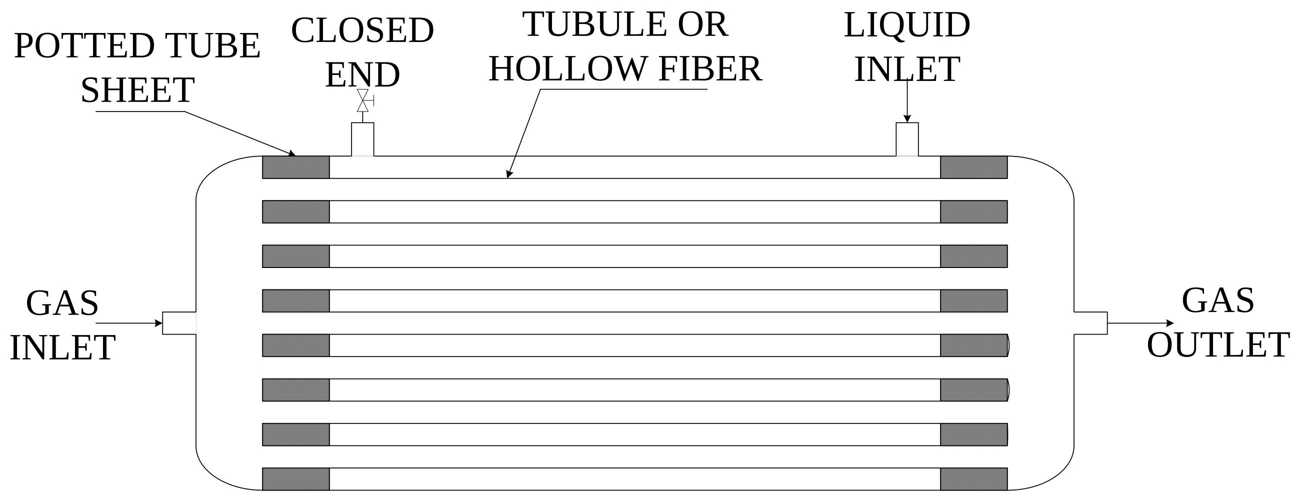
Concentration profile for absorbed species in gas and liquid phases in a membrane contactor



Schematic of the absorber containing ceramic tubules or hollow fibers



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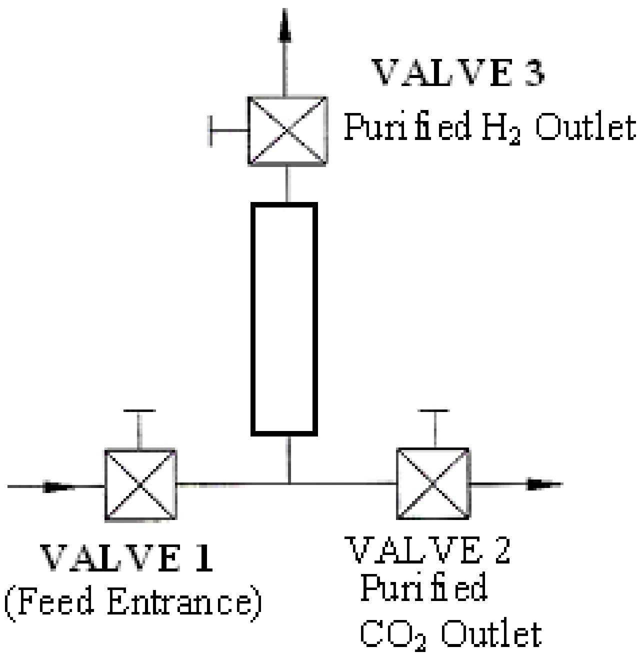
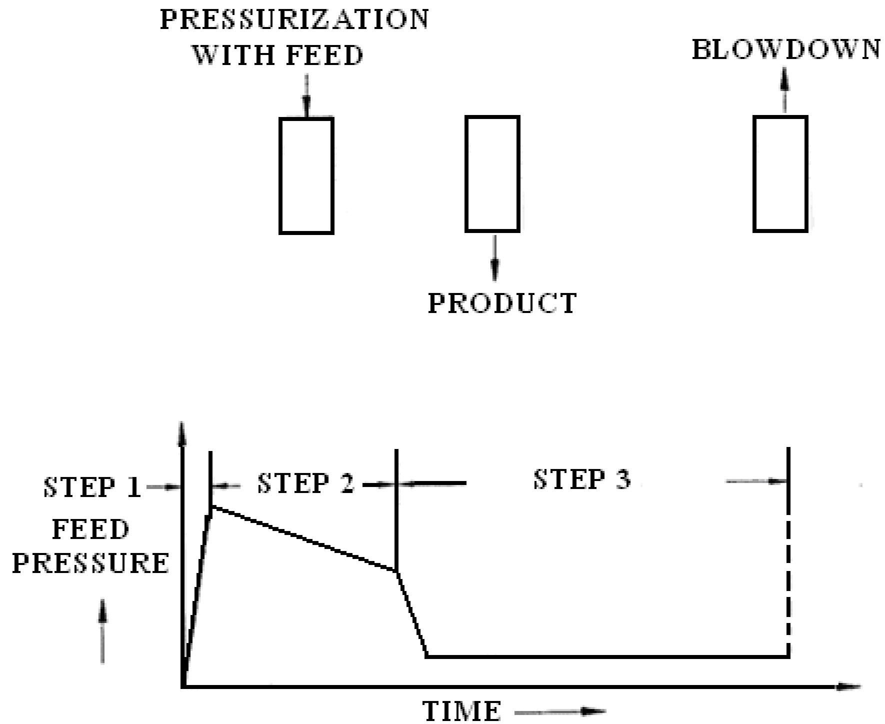
Pressure Swing Absorption Operation



Solenoid Valve Locations in Pressure Swing Absorption (PSAB) Apparatus



Pressure vs. time profile in the bore of tubule or hollow fiber in PSAB

(Bhaumik et al., *AIChE J.*, 42, 409-421 (1996))

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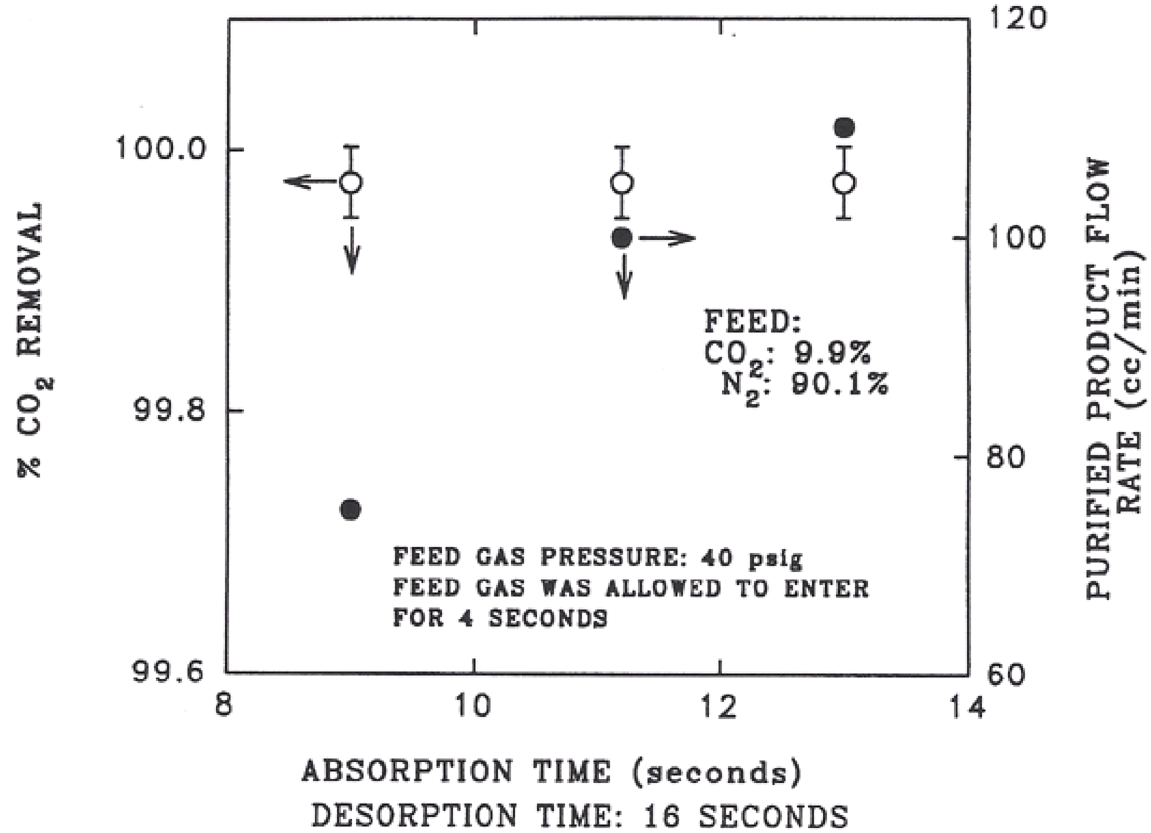
Pressure Swing Absorption (PSAB) in a Membrane Contactor Device

- Basic separation concept implemented with 10% CO_2-90% N_2 gas mixture at 375 kPa and 19.5 wt% aqueous DEA solution (RAPSAB – Rapid Pressure Swing Absorption)

- Its adaptation to the current problem of treating low temperature post-shift reactor synthesis gas at ~20 atm and 150-200^{0}C

Experimental results for RAPSAB using DEA solution as an absorbent



(Bhaumik et al., *AIChE J.*, 42, 409-421 (1996))

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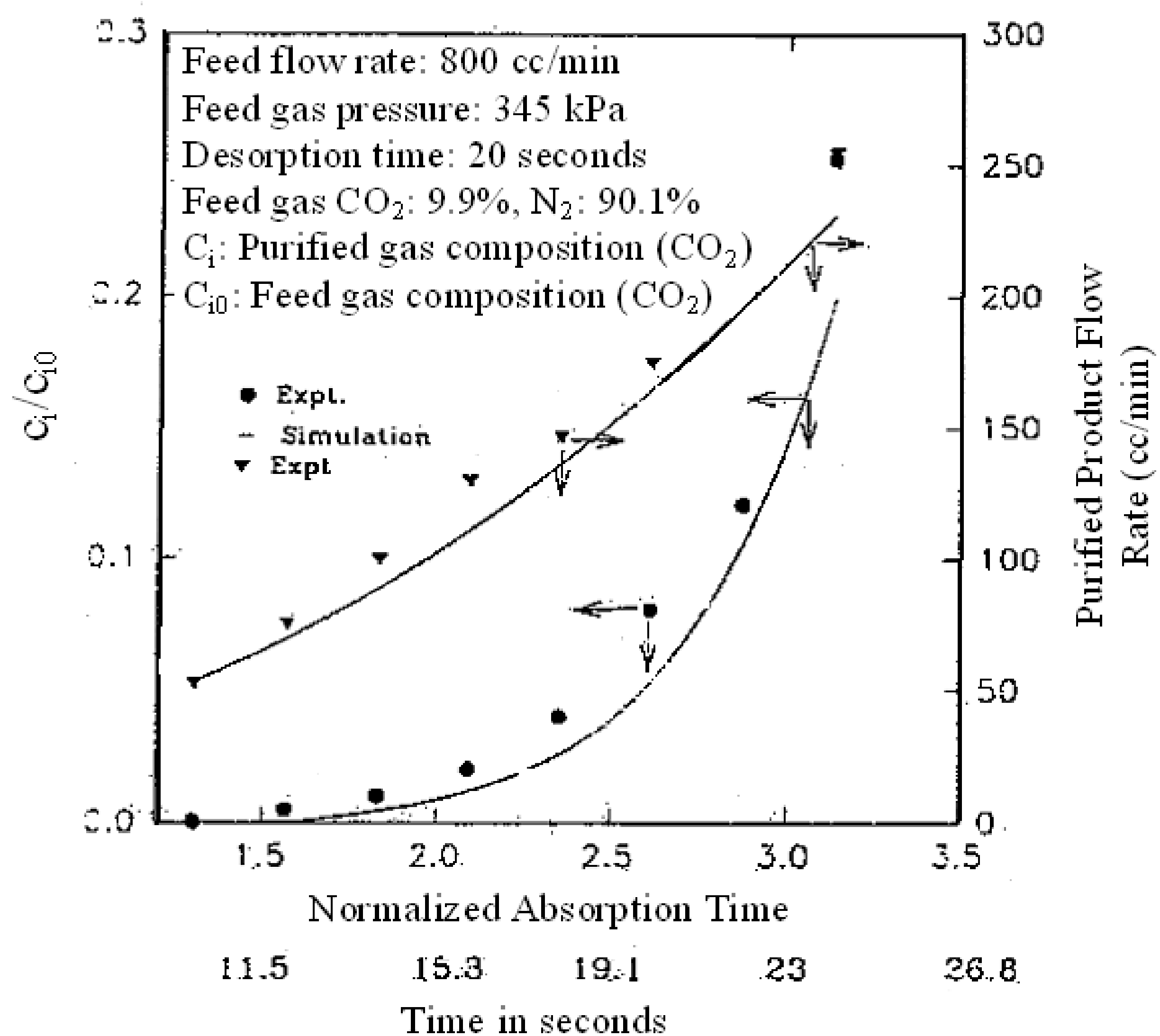
Removal of CO_2 with DEA as an absorbent: experimental results vs. theoretical simulations



Potential Advantages of the Proposed Separation Technique-1

- Has high solubility selectivity of novel selected liquid absorbents having relatively high viscosity
- Has high purification ability of pressure swing adsorption process
- Has high gas-liquid contacting surface area per unit device volume
- Has a compact membrane-like device
- Scale up should be easier due to modularity of membrane-based devices and membrane-based phase contacting

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Potential Advantages of the Proposed Separation Technique-2

- Will deliver highly purified H_2 at nearly its partial pressure and temperature in the post-shifted reactor synthesis gas feed

- Purified CO_2 stream (>90% CO_2) will be available at 1-5 atm

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Nonvolatile Absorbents for PSAB

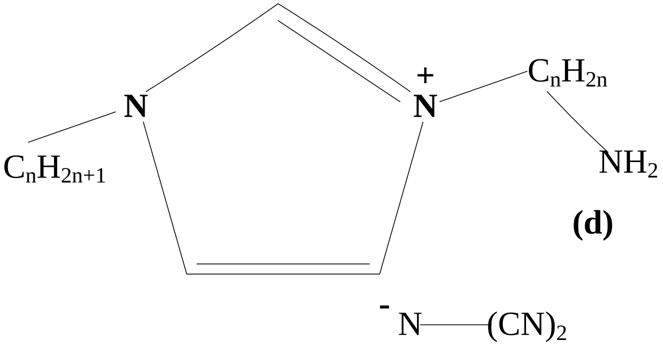
1. Ionic Liquids:

$[bmim]^+[PF_6]^-$ (Fluka)

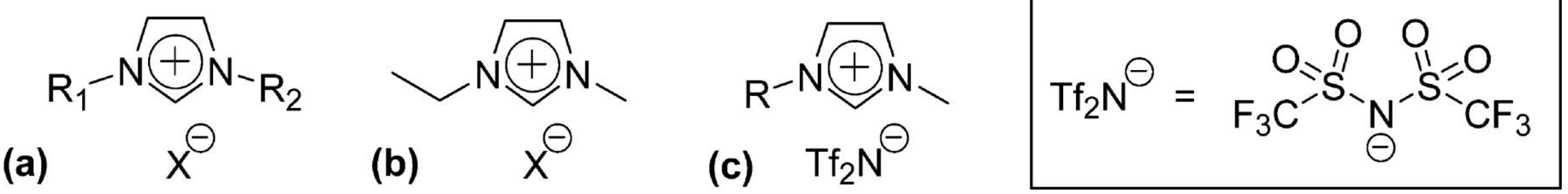
$[bmim]^+[DCA]^-$ (Merck)

$[Am\text{-}Im]^+[DCA]^-$ (Functionalized)

(with or without moisture)



(d) Functionalized IL structure for $[Am\text{-}Im]^+[DCA]^-$



General structures of (a) imidazolium-based RTILs, (b) $[C_2mim][X]$ RTILs, and (c) $[Rmim][Tf_2N]$ RTILs. (Bara et al., *Ind. Eng. Chem. Res.*, 48, 2739 (2009))

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Nonvolatile Absorbents for PSAB

2.(a) Dendrimers/hyperbranched polymers of lower molecular weight:

Polyamidoamine (PAMAM) generation 0, MW-516, 4 primary amines, 2 tertiary amines;

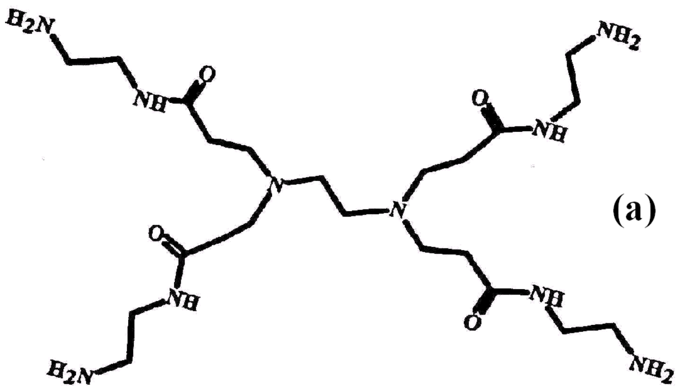
Generation 2, MW-3130

(Dendritech, Midland, MI)

Use in a nonvolatile solvent,

such as polyethylene glycol (PEG 400)

Highly reactive in the presence of moisture



(a) PAMAM dendrimer of generation 0

(1. Kovvali et al., *JACS*, 122 (31) 7594 (2000); 2. Kovvali and Sirkar, *I&E C Res.*, 40(11), 2502 (2001); 3. Kosaraju et al., *I&E C Res.*, 49, 1250 (2005))

(b) Polyethyleneimines of lower molecular weight, Lupasol FG (BASF), MW-615

(Rolker et al., *I&E C Res.*, 46, 6572 (2007))

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Adaptation of PSAB Device to the Current Problem

1. Porous PP membrane substrate replaced by hydrophobized porous ceramic tubules, porous PTFE hollow fibers and hydrophobized PEEK hollow fibers (higher temperature, wettability considerations)
2. High pressure means smaller membrane pore size (5 nm to 10 nm) at the gas-liquid interface to prevent any phase breakthrough
3. Longer length of hollow fibers in RAPSAB replaced by number of ceramic tubules in series (limitation of oven dimensions)

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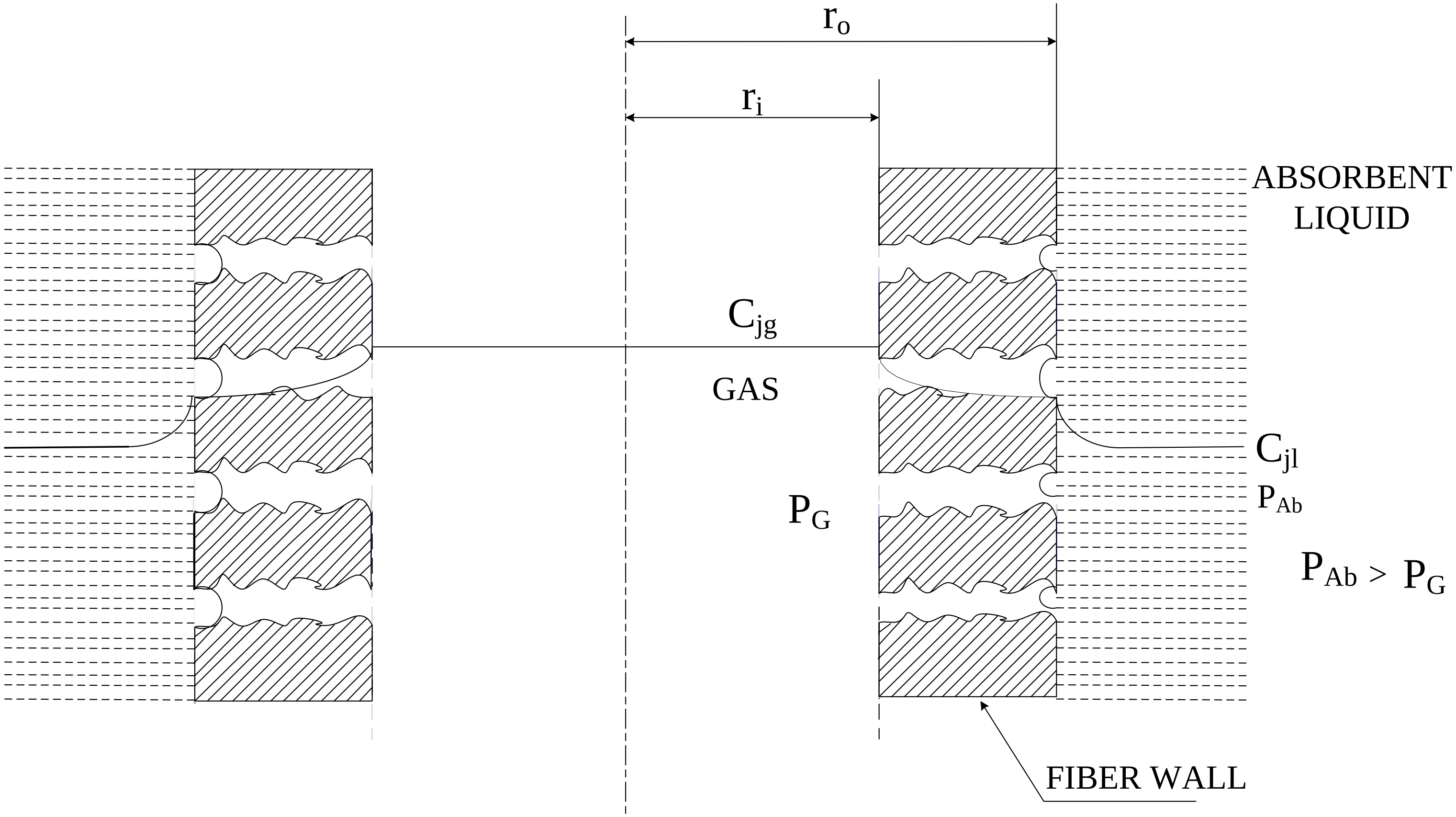
Concentration profile for absorbed species in gas and liquid phases



Breakthrough pressure for a nonwetted pore size of radius r_p (Young-Laplace Equation)

$$\Delta P_{breakthrough} \cong \frac{2\gamma \cos\theta}{r_p}$$

Increase γ , decrease r_p

The pores should remain nonwetted.

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Nondisperssive Gas Absorption/Stripping Requires Nonwetted Pores

1. To prevent spontaneous pore wetting

Surface tension of absorbent liquid

$\gamma > \gamma_{critical}$ of the polymeric coating

2. $\gamma_{critical}$ of fluoropolymers, C_{18} surfaces….15-20 dyne/cm
3. Absorbent liquids under consideration have considerably higher γ values; γ will fluctuate due to absorption and desorption of moisture

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Surface Tension / Interfacial Tension

1. $$\gamma = \gamma_{20^0C}\left(\rho \Big/ \rho_{20^0C}\right)^4$$

   Hasse et al., *J. Chem. Eng. Data*, <u>54</u>, 2576 (2009).

   $[EMIM][MeOHP_2]$ etc.

   Density decreased by about 0.05-0.1 gm/cm^3 over 100^{0}C; density in the range of 1.05-1.35 gm/cm^3

2. Galan Sanchez et al., Trans. I. Chem. E.,Part A, *Chem. Engg. Res. & Dev*., <u>85</u> (A1), 31 (2007)

   →40-45 mN/m for $[bmim]^+[BF_4]^-$

3. Klomfar et al., *J. Chem. & Engg. Data*, <u>54</u>, 1389 (2009)
   $[C_nmim][PF_6]$ → γ decreasing from around 50 mN/m for C_3 to 36 mN/m at C_8

4. PAMAM dendrimer of generation 0 →55 mN/m at 25^{0}C (Kosaraju et. Al., *I & E C Res*., <u>44</u>, 1250 (2005)

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- **Ceramic Tubules:**

  1.5 mm I.D., 3.8 mm O.D. γ-alumina coating on alpha-alumina substrate hydrophobized with nonafluorohexylsilane coating

  Pore radius ~5 nm <0.03µm , 940 m^2/m^3 surface area/device volume

  For say, a 40 dyne/cm liquid to withstand 20 atm+, 10 nm pore size

  C_{18} hydrophobic coatings and epoxy-based tube sheet up to 200^0C

(Media and Process Technology, Pittsburgh, PA; Rich Ciora/Paul K.T. Liu)

- **Teflon Hollow Fibers:**

  0.53 mm I.D., 1.08 mm O.D. Pore size <0.1µm

  Plasma polymerize a nanoporous fluorosilicone coating to reduce the pore size to ≤0.01µm

(Applied Membrane Technologies, Inc., Minnetonka, MN; Stephen Conover)

- **PEEK Hollow Fibers:**

  300 µm I.D., 500 µm O.D., porous hollow fibers of poly (ether ether ketone), hydrophobized surface via fluorination of the surfaces

(Porogen Inc., Woburn, MA; Ben Bikson)

M&P ceramic membrane tubes: 2″ and 4″ commercial elements

CO_2 Gas-Liquid/Liquid-Gas Mass Transfer Aspects

- Stagnant highly viscous absorbent liquid on the shell side
- Tube-side flowing gas present in pores of membrane

$$Rate\ of\ physical\ gas\ absorption \quad \alpha \quad \sqrt{\frac{D_{CO_2}}{\pi t}}$$

$$Amount\ absorbed\ per\ unit\ area \quad \alpha \quad \sqrt{\frac{D_{CO_2} t}{\pi}}$$

$$D_{CO_2} \quad \alpha \quad \frac{1}{\mu_{absorbent}}$$

- High temperature of operation will reduce $\mu_{absorbent}$ drastically

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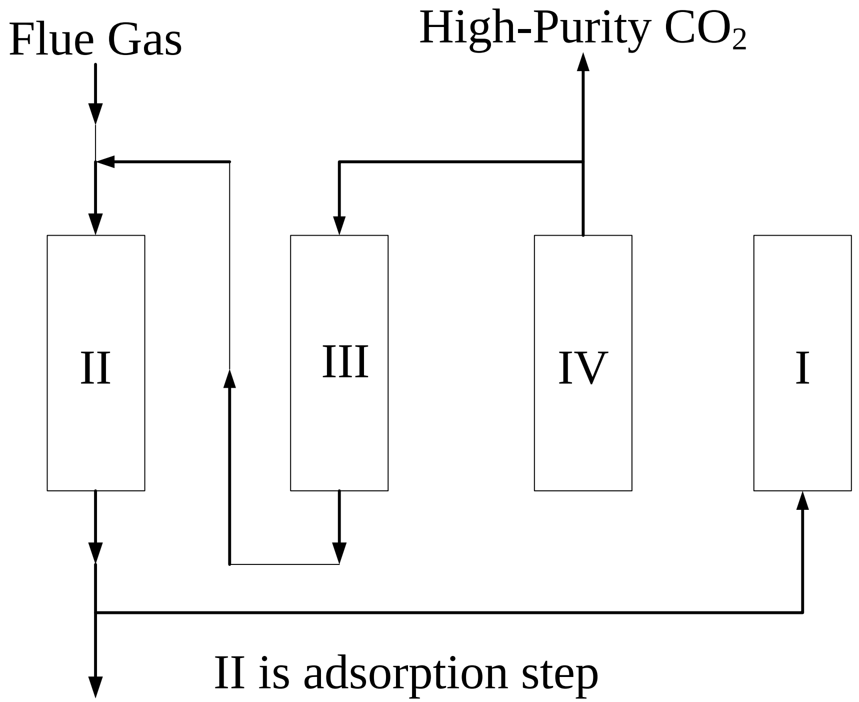
Gas Mixture to be Studied

- 45% He, 30% CO_2, Rest being H_2O
- 150-200^{0}C, 200-300 psig
- Helium as a surrogate for H_2

- Typical Gasifier Composition:

  ~38% H_2, 29% CO_2, 33% H_2O, 0.15% CO

Concentration and Recovery of CO_2 from Flue Gas by Pressure Swing Adsorption



II is adsorption step

III is purge with concentrated CO_2 step

IV is countercurrent blowdown

I is pressurization with adsorption product step

(E.S. Kikkinides, R.T.Yang and S.H.Cho, *Ind. Eng. Chem. Res.*, 32, 2714 (1993))

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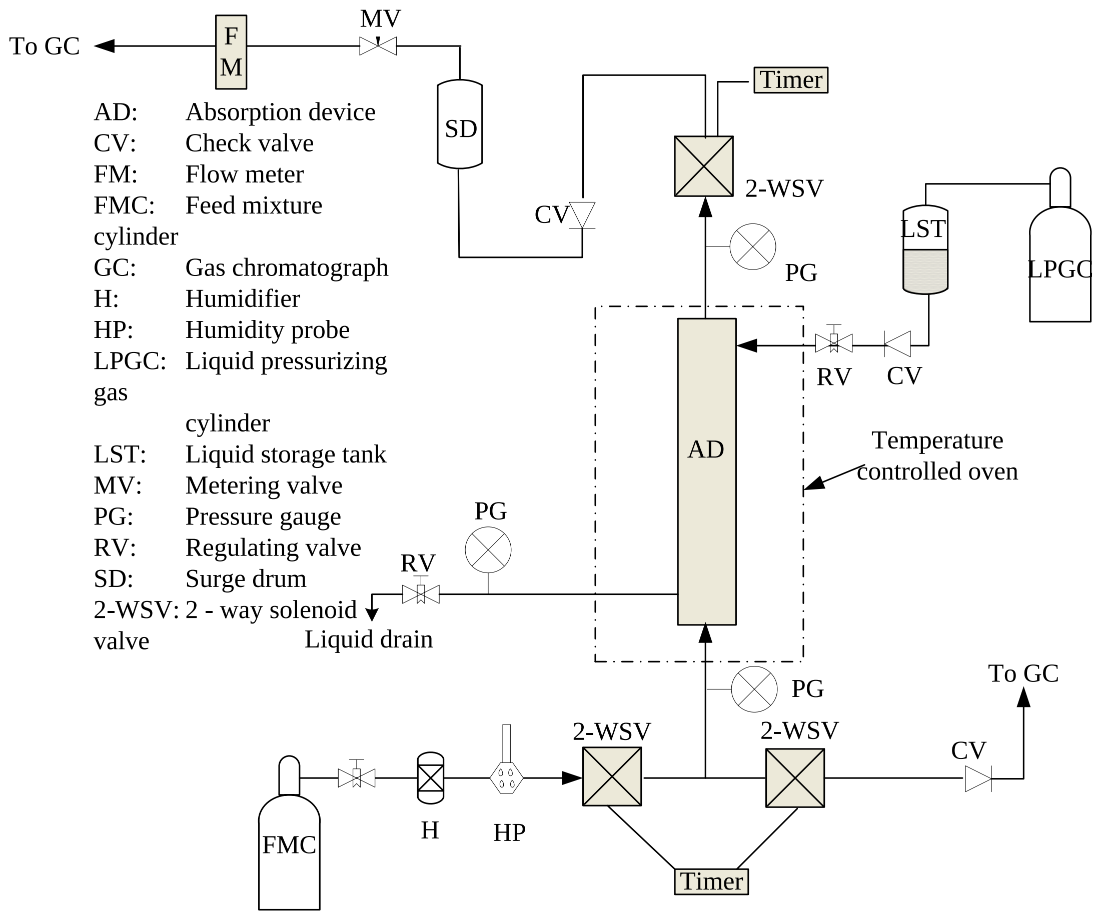
Schematic of the experimental setup for pressure swing absorption



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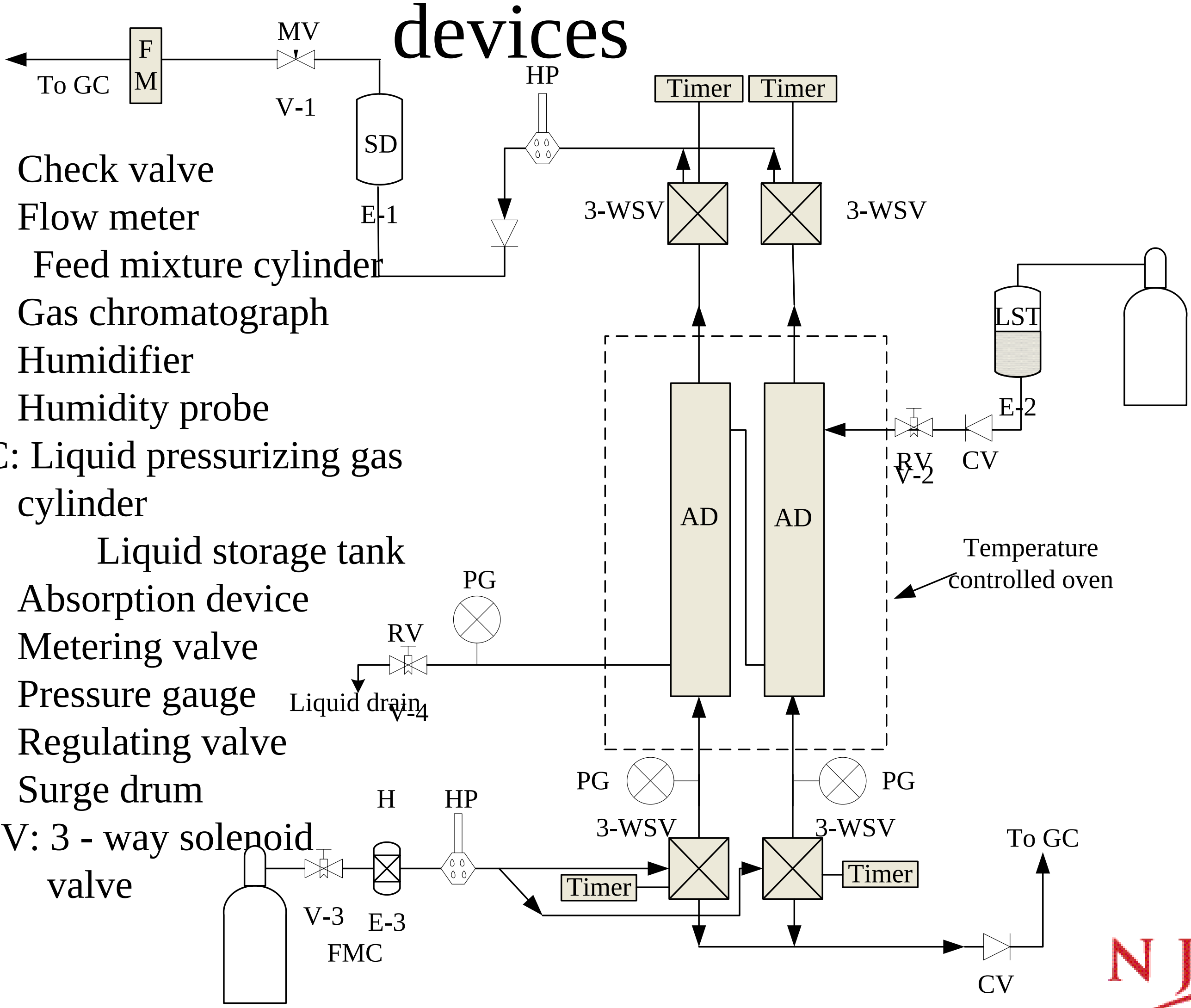
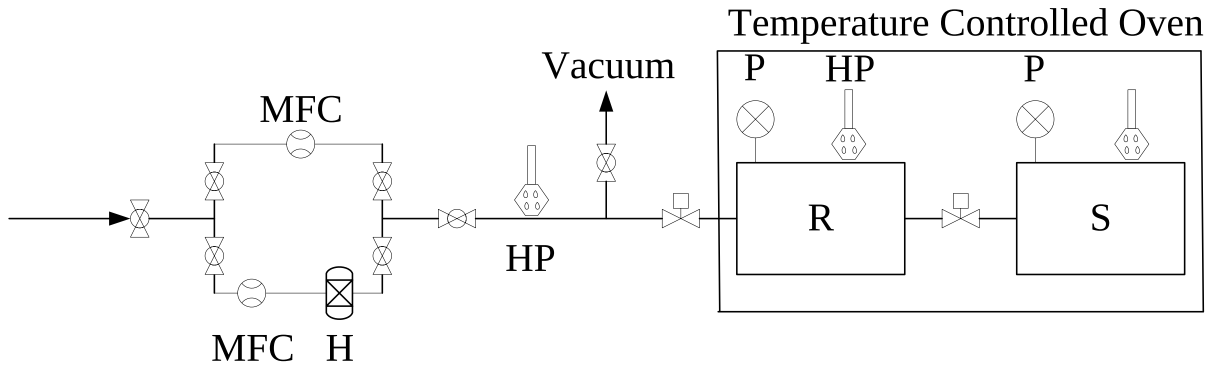
Schematic of the experimental setup for pressure swing absorption using two separate absorption devices

CV: Check valve
FM: Flow meter
FMC: Feed mixture cylinder
GC: Gas chromatograph
H: Humidifier
HP: Humidity probe
LPGC: Liquid pressurizing gas cylinder
LST: Liquid storage tank
AD: Absorption device
MV: Metering valve
PG: Pressure gauge
RV: Regulating valve
SD: Surge drum
3-WSV: 3 - way solenoid valve



Schematic of the measurement of gas uptake by an absorbent liquid



H Humidifier
HP Humidity probe
MFC Mass flow controller
P Pressure transducer

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Project Objectives

- Develop via laboratory experiments an advanced pressure swing absorption-based device and a cyclic process to produce purified helium (a surrogate for hydrogen) at a high pressure for IGCC-CCS plant's combustion turbine from low temperature post-shift reactor synthesis gas and simultaneously obtain a highly purified CO_2 stream containing at least 90% of the CO_2 in the post-shift reactor gas stream and suitable for subsequent sequestration

- Provide data and analysis of the cyclic process and device to facilitate subsequent scale up

- Develop a detailed analysis for the process and device to allow economic evaluation for potential larger-scale use

Project Objectives: PHASE-I

I1. Develop an experimental setup for studying the PSAB process

I2. Develop novel gas-liquid absorption modules employing ceramic tubules and polymeric hollow fibers of PTFE

I3. Initiate preliminary studies of pressure swing absorption-based separation of a moist CO_2-He gas mixture at 150-200^{0}C and 200-300 psig simulating a low temperature post-shift reactor synthesis gas stream

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Project Objectives: PHASE-II

II1. Study the performance of the PSAB process for selected absorbents vis-à-vis purification of the feed gas stream to obtain a high pressure purified He stream and a low pressure purified CO_2 stream

II2. Develop experimental setups to measure the solubility and diffusion coefficients of CO_2 and He at the appropriate ranges of temperature and pressure for selected absorbents

II3. Initiate development of a mathematical model of the PSAB device and process

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Project Objectives: PHASE-III

III1. Generate experimental data on the solubility and diffusion coefficient for CO_2 and He for the selected absorbents

III2. Compare the results of simulation of the mathematical model with the observed purification and separation in the PSAB process and device for selected absorbents

III3. Perform simulations of the model to explore scale up of the process to facilitate evaluation of the process

III4. Determine the extent of loss/deterioration of the absorbents over extended periods of operation

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Phase I Progress
Tasks to be Performed: Phase I

- Task 1.0 Project Management and Planning (10/1/09 – 9/30/10)
- Subtask 1.1 Provide quarterly reports at the end of every quarter as well as a Topical Report at the end of year 1 (10/1/09 – 9/30/10)
- Task 2.0 Experimental Program and Technical Activities for Year 1 (10/1/09 – 9/30/10)
- Subtask 2.1: Develop an experimental setup for studying the PSAB device and process (10/1/09 – 8/31/10)
- Subtask 2.2: Develop novel gas-liquid absorption modules (10/1/09 – 9/30/10)
- Subtask 2.3: Initiate preliminary studies of PSAB process (8/1/10 – 9/30/10)

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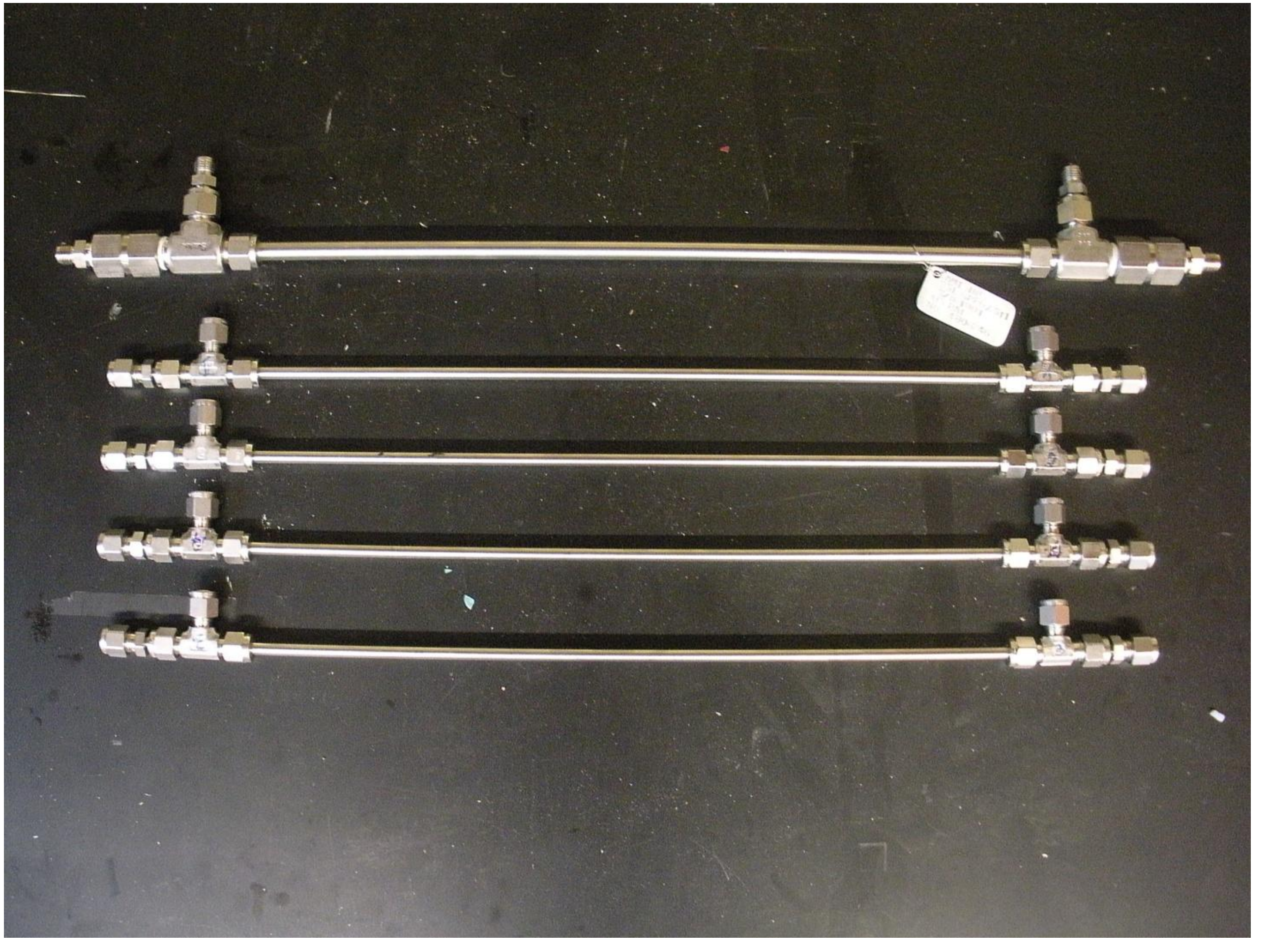
Details of Membrane Modules

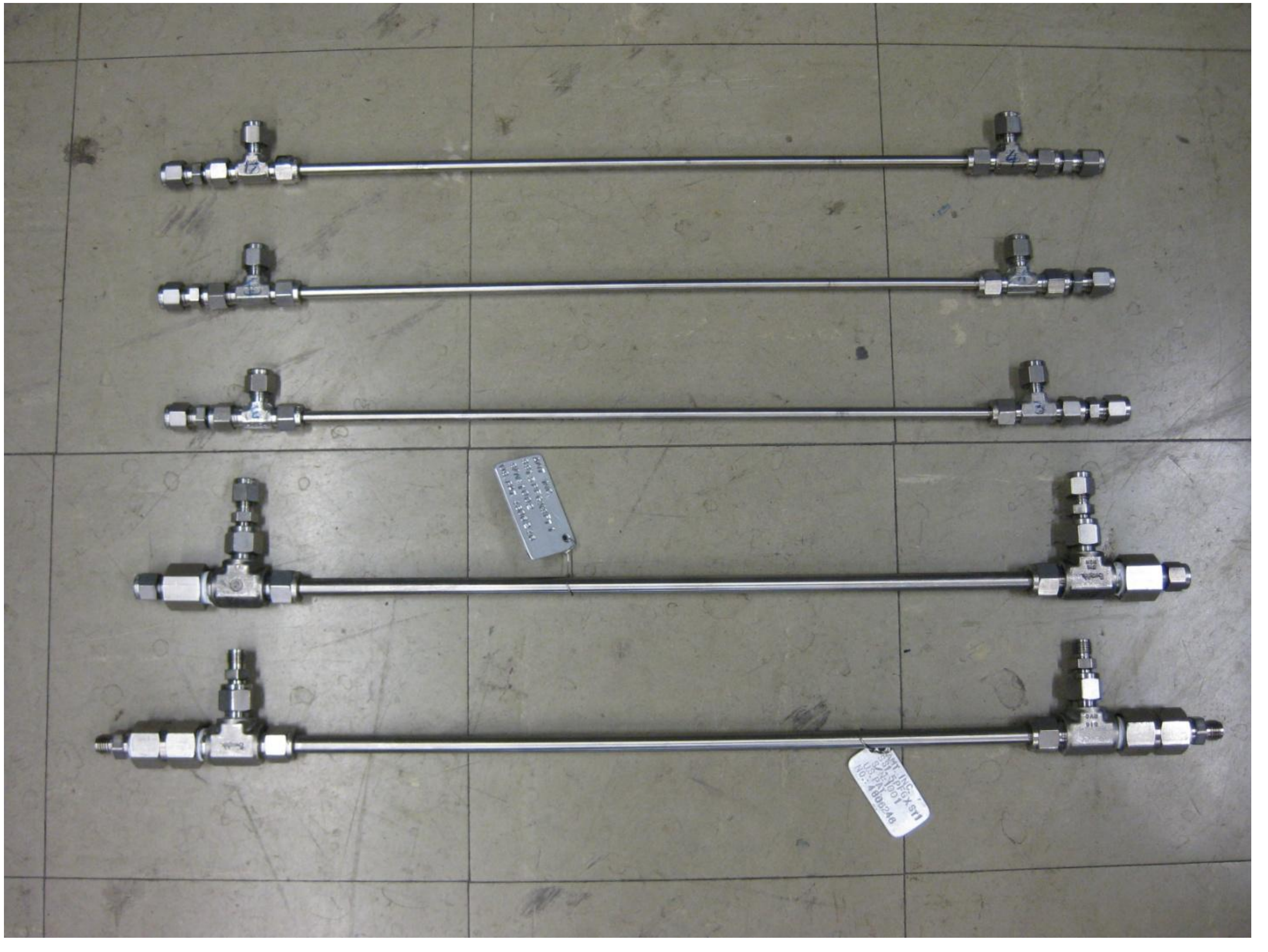
Membrane module type	Details of Membrane Modules					
	I.D. mm	O.D. mm	Pore Size µm	No. of fibers/tubules	Length	Shell Diameter
*Ceramic membrane modules # 1,2,3,4	1.5	3.8	0.005	1	18”	1/4 “ OD 0.035” wall thickness
Teflon hollow fiber module	0.53	1.08	~0.01	18	18”	3/8” OD .035” wall thickness
PEEK hollow fiber module	0.25	0.45	~0.01		18”	¼”

* Single ceramic tubule, ‘o’-ring seal.

Module with Teflon Hollow Fibers and 4 Modules containing Ceramic Membrane Tubules



Ceramic Membrane Tubules in Modules and Teflon Modules



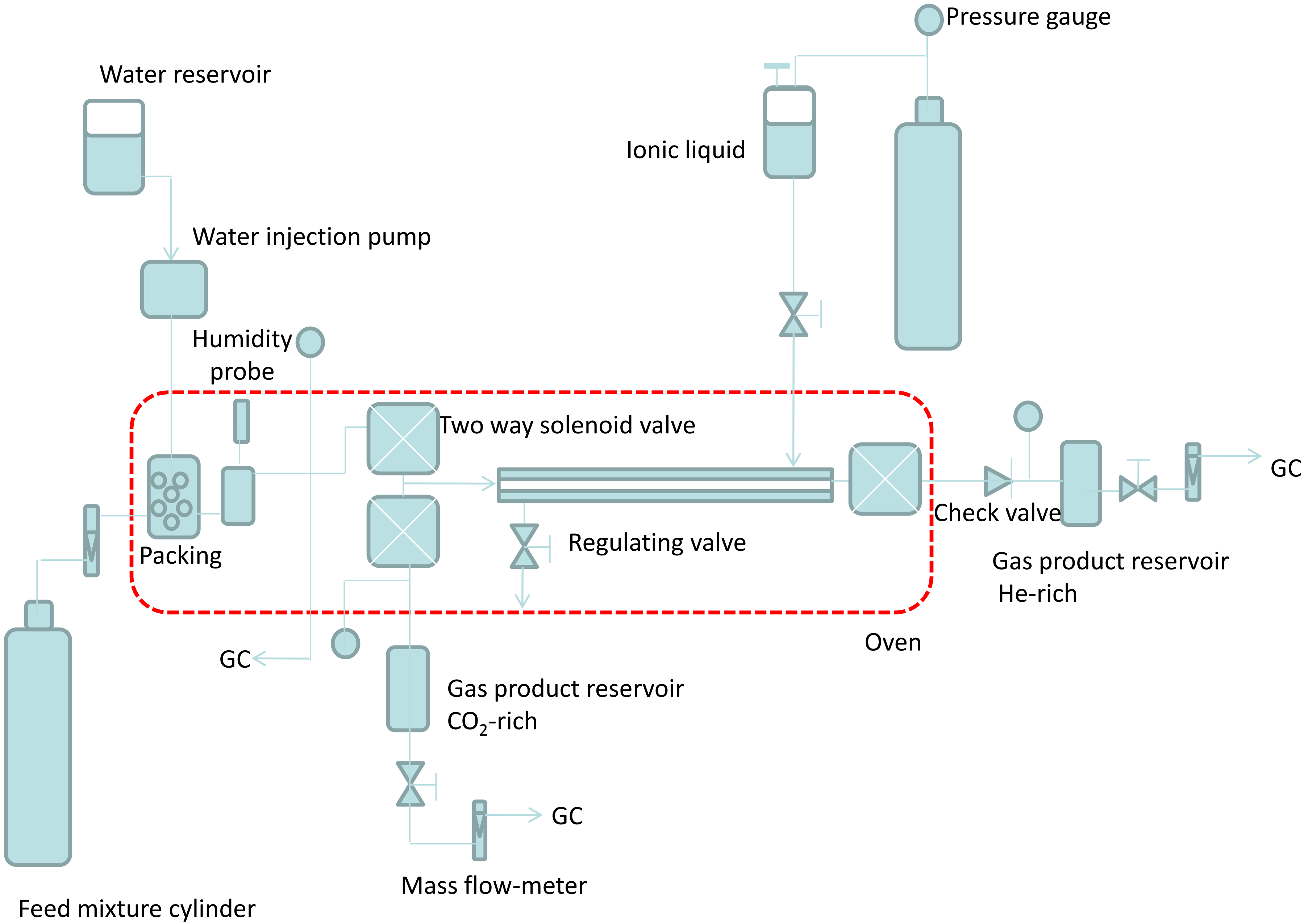
Liquid Breakthrough Pressure test results
for 4 ceramic membrane modules and Teflon hollow fiber modules

Module # \ Liquid Tested	Water	Glycerol Carbonate	[Bmim][DCA]	[Emim][Tf_2N]
*Ceramic 1	-	No leakage up to 260 psi	No leakage up to 260 psi	160 psi
*Ceramic 2	150 psi	80 psi	NA	NA
*Ceramic 3	This module may be broken	NA	NA	NA
*Ceramic 4	NA	150 psi	80 psi	NA
**Teflon	No leakage up to 80 psi	No leakage up to 80 psi	NA	NA

* Single ceramic tubule; ** Tests are being done at higher pressures.
Tests were carried out at room temperature.

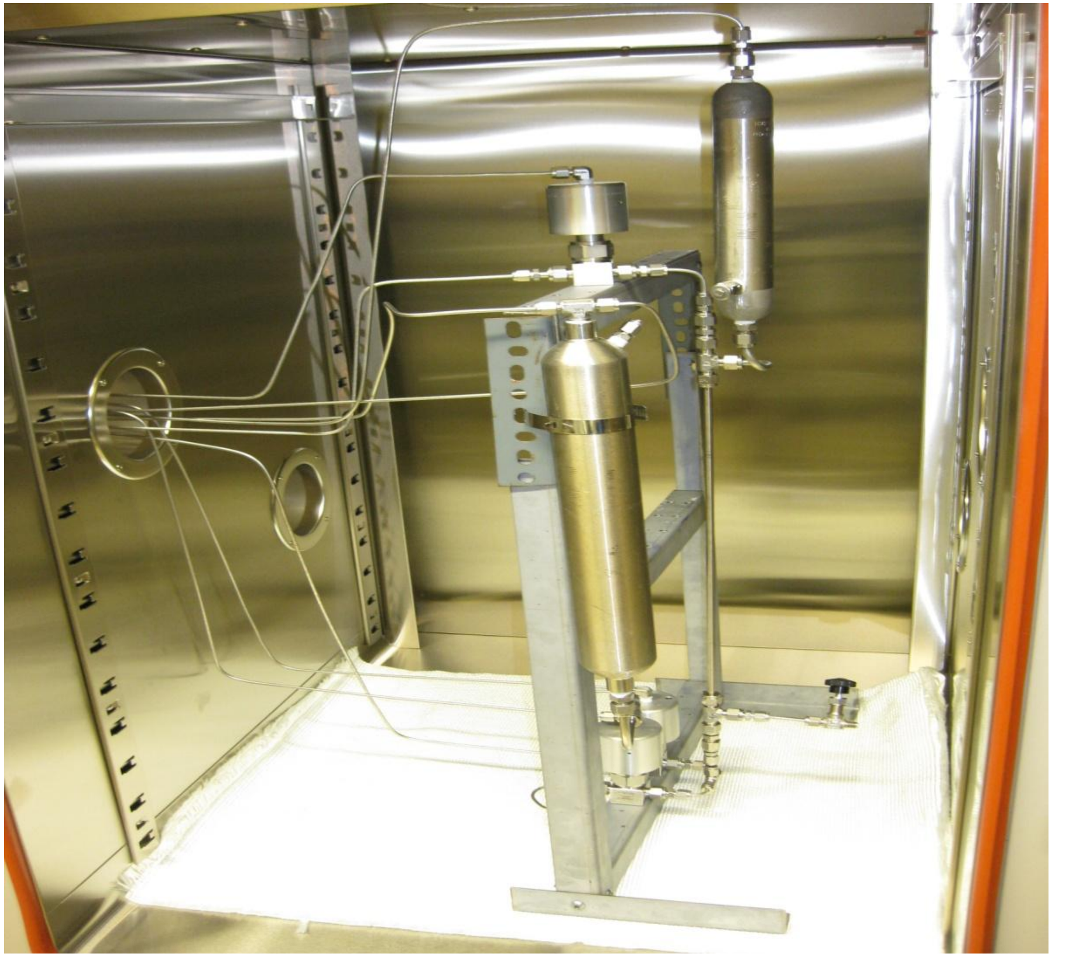
Surface tension: Water, 72.8mN/m; Glycerol Carbonate, 45.5mN/m; [Bmim][DCA], 48.5mN/m; [Emim][Tf_2N], 41.6mN/m

ESPEC Oven Interior

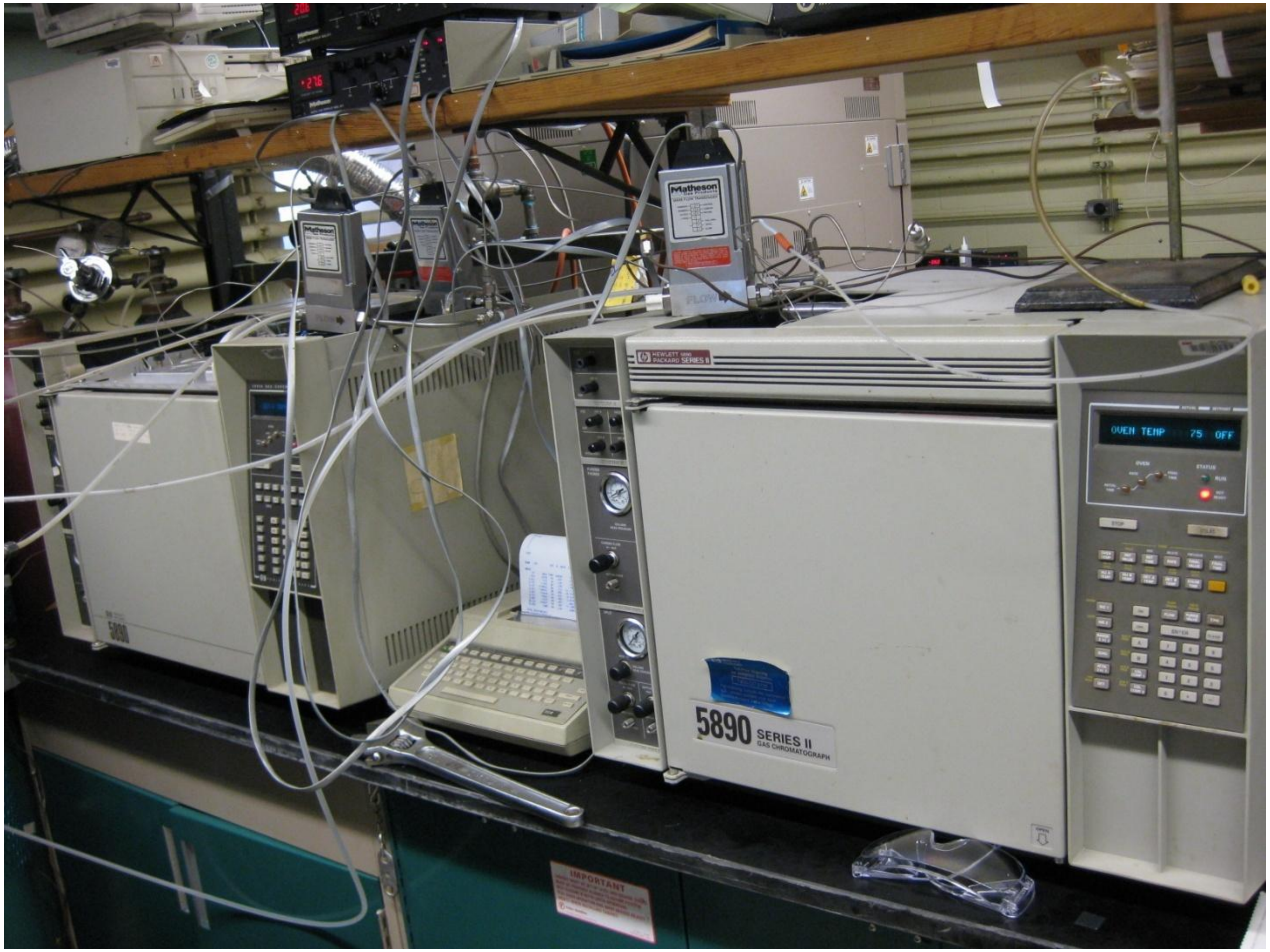


Pressure gauge
Water reservoir
Ionic liquid
Water injection pump
Humidity probe
Two way solenoid valve
Check valve
Packing
Regulating valve
GC
Gas product reservoir He-rich
Oven
GC
Gas product reservoir
CO_2-rich
GC
Mass flow-meter
Feed mixture cylinder

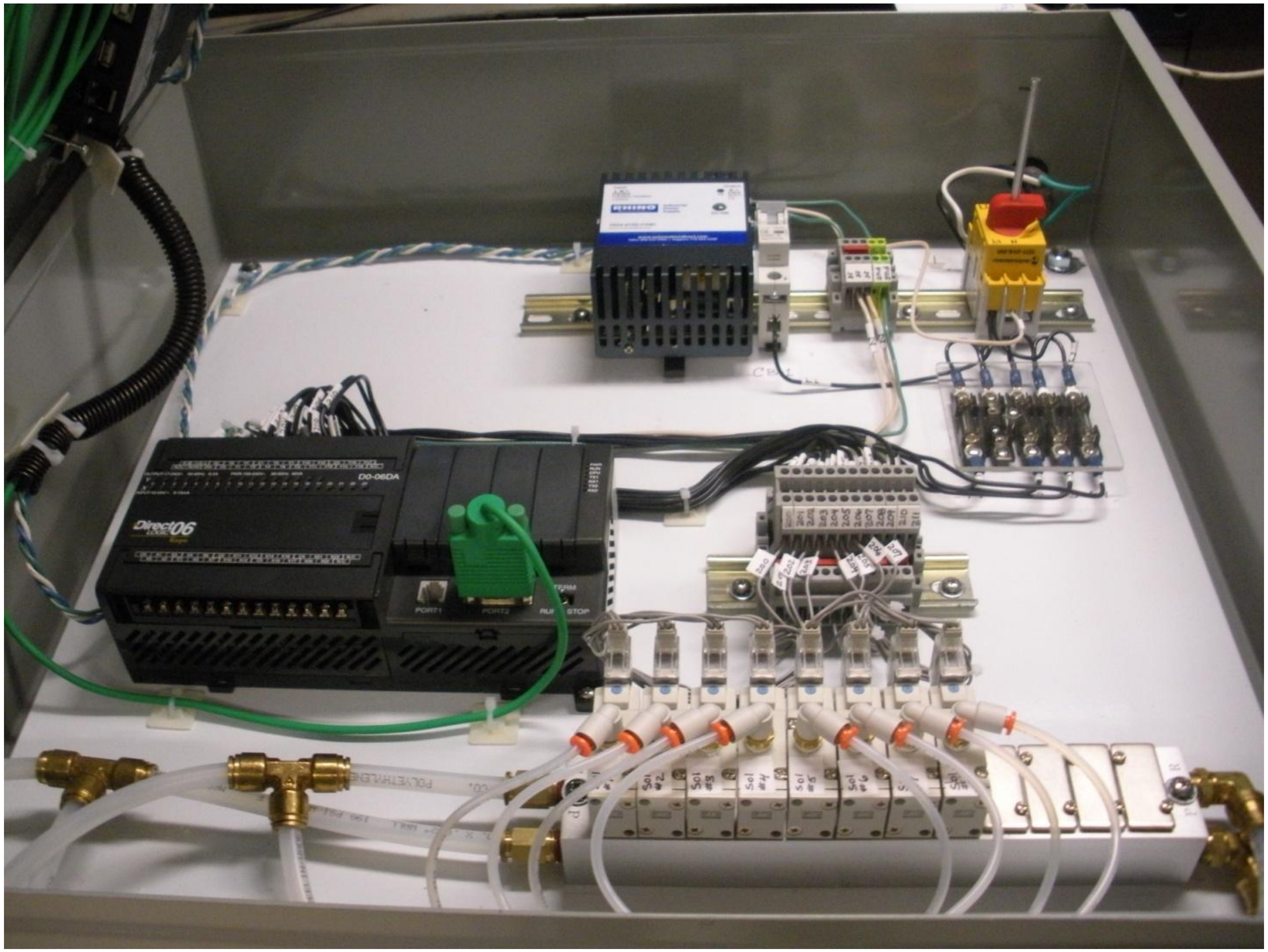
Experimental Setup inside the Oven for PSAB



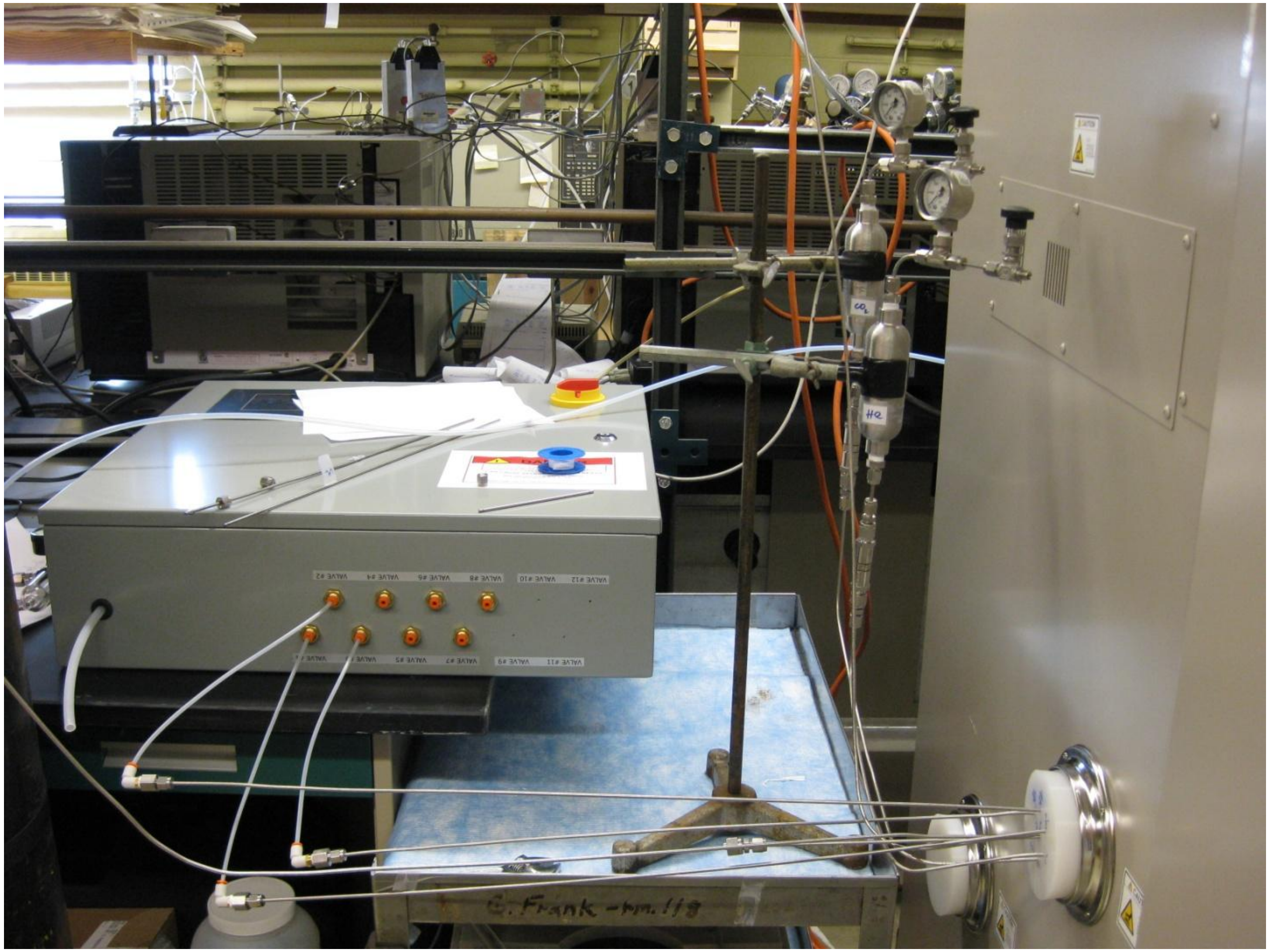
GCs



PneuMagnetic PLC Control Box



PneuMagnetic PLC Control Box with Connections to the Valves inside the Oven



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Project Structure

- Project Structure may be described through the List of Tasks describing in detail the following steps
- Design the device and PSAB process after selecting absorbents and the dimensions of the membrane units
- Build the setup
- Perform separation runs
- Analyze the data and focus on conditions showing the desired performance
- Develop a mathematical model for the process
- Determine the solubility and diffusivity of solutes in the absorbent liquids
- Compare model results with experimental data
- Determine absorbent deterioration with time

Tasks to be Performed: Phase II

- Task 3.0 Project Management and Planning (10/1/10 – 9/30/11)
- Subtask 3.1 Provide quarterly reports at the end of every quarter as well as a Topical Report at the end of year 2 (10/1/10 – 9/30/11)
- Task 4.0 Experimental Program and Technical Activities for Year 2 (10/1/10 – 9/30/11)
- Subtask 4.1 Study the performance of PSAB devices and the PSAB process (10/1/10 – 9/30/11)
- Subtask 4.2 Develop experimental setups to measure solubility and diffusion coefficients of CO_2 and He in selected absorbent liquids (10/1/10 – 9/30/11)
- Subtask 4.3 Initiate development of a mathematical model of the PSAB device and process (10/1/10 – 9/30/11)

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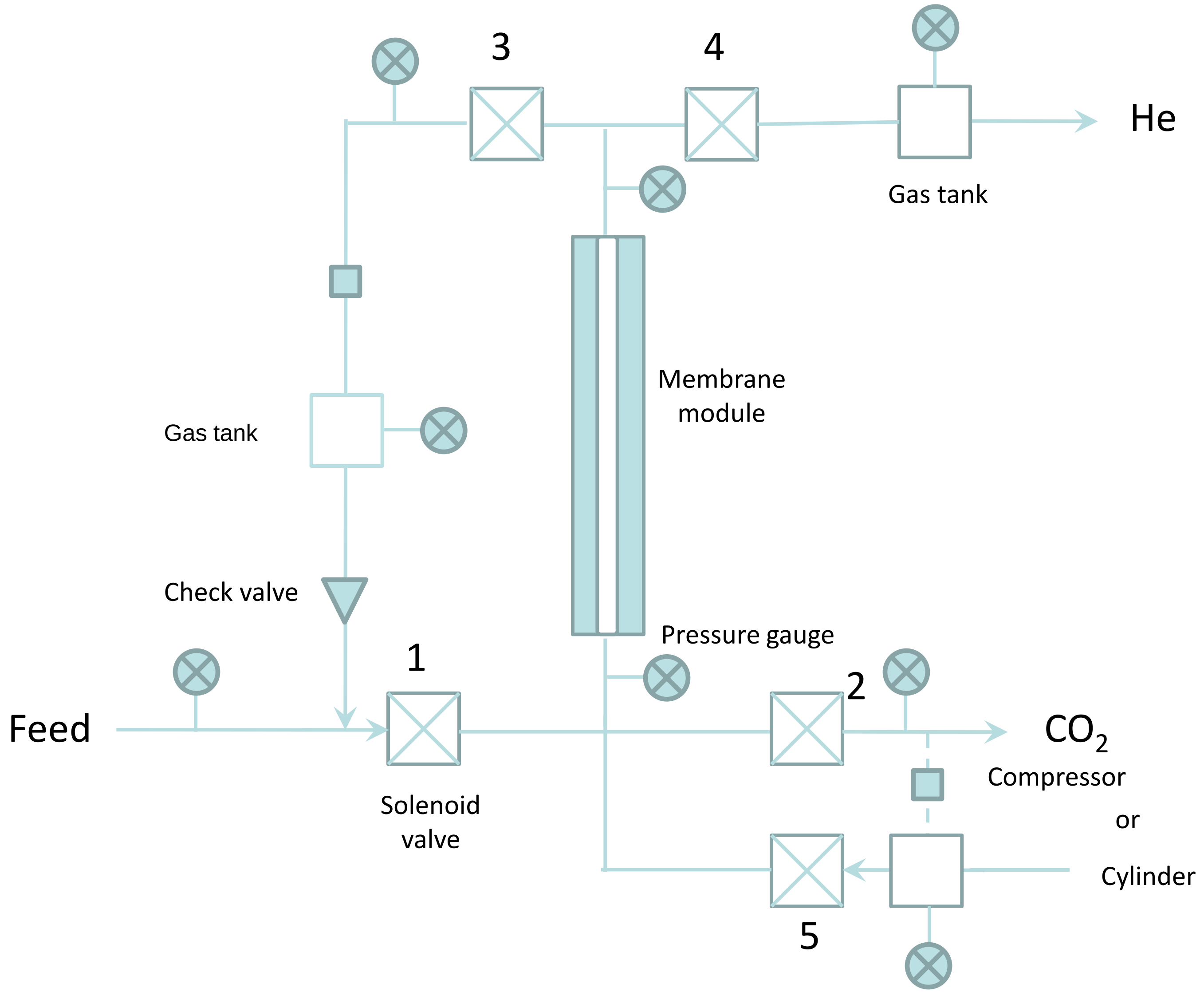
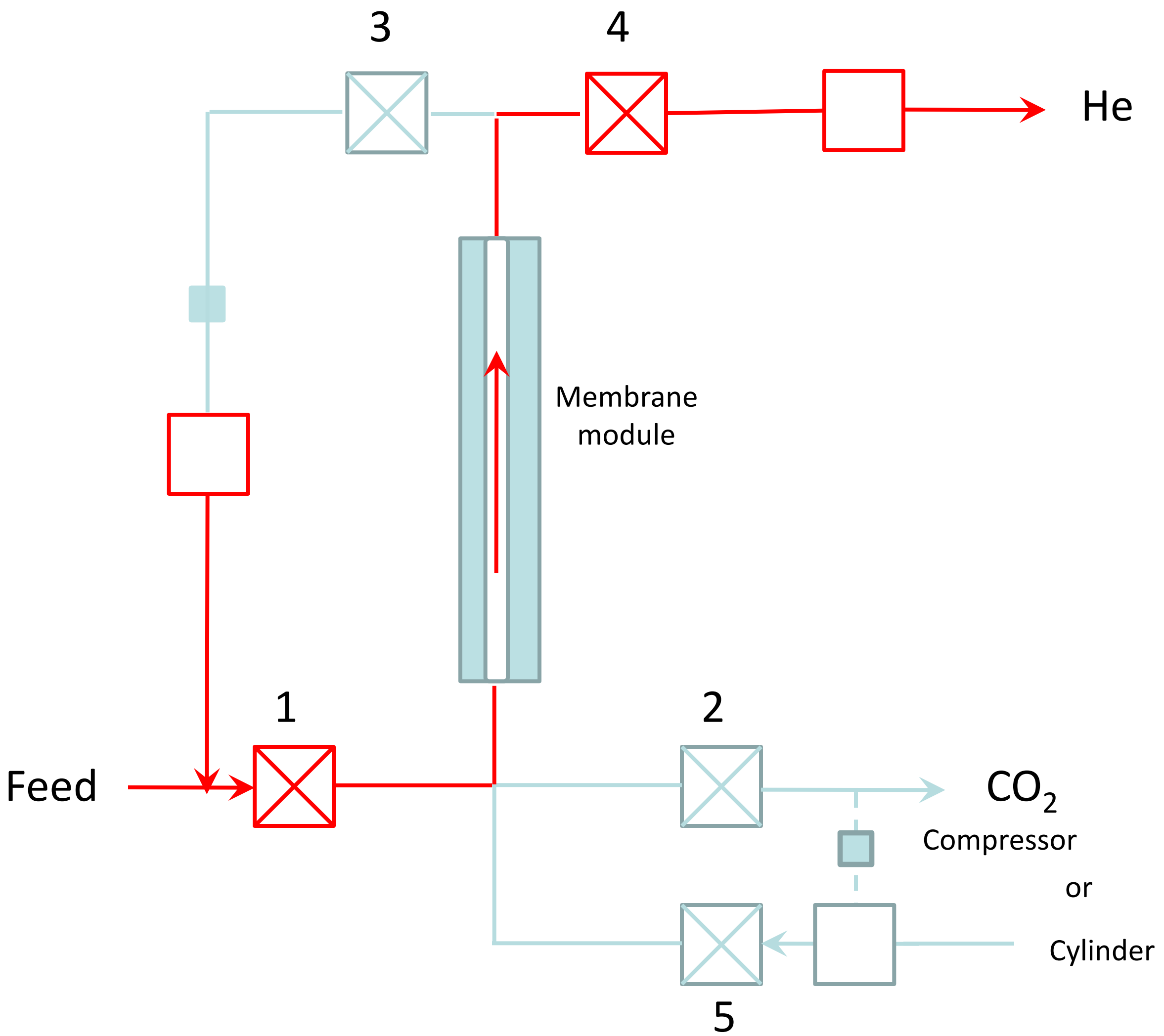
One membrane module cycle

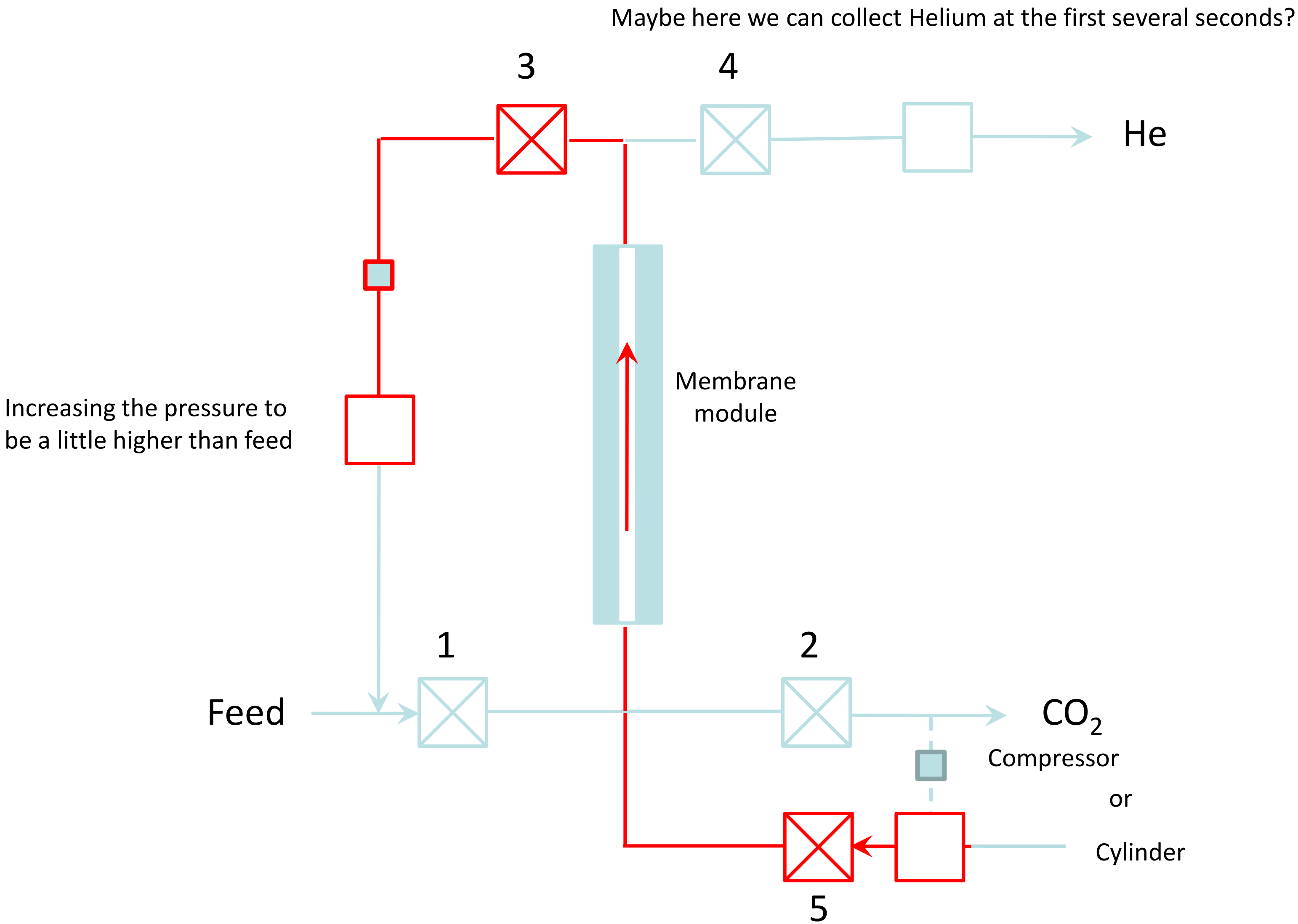


Step 1 Feed in and Absorption

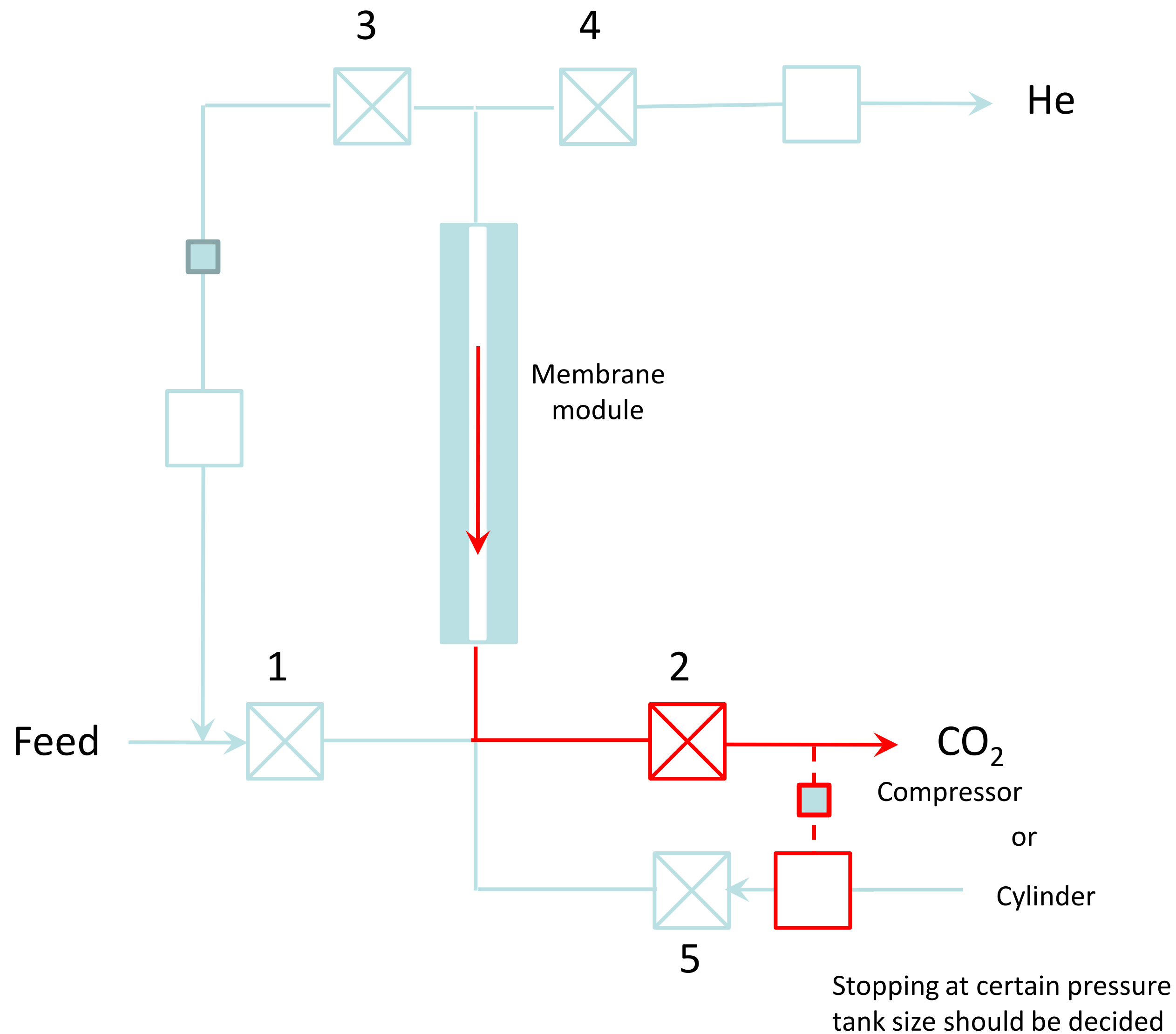


Red color means the opened line and valves

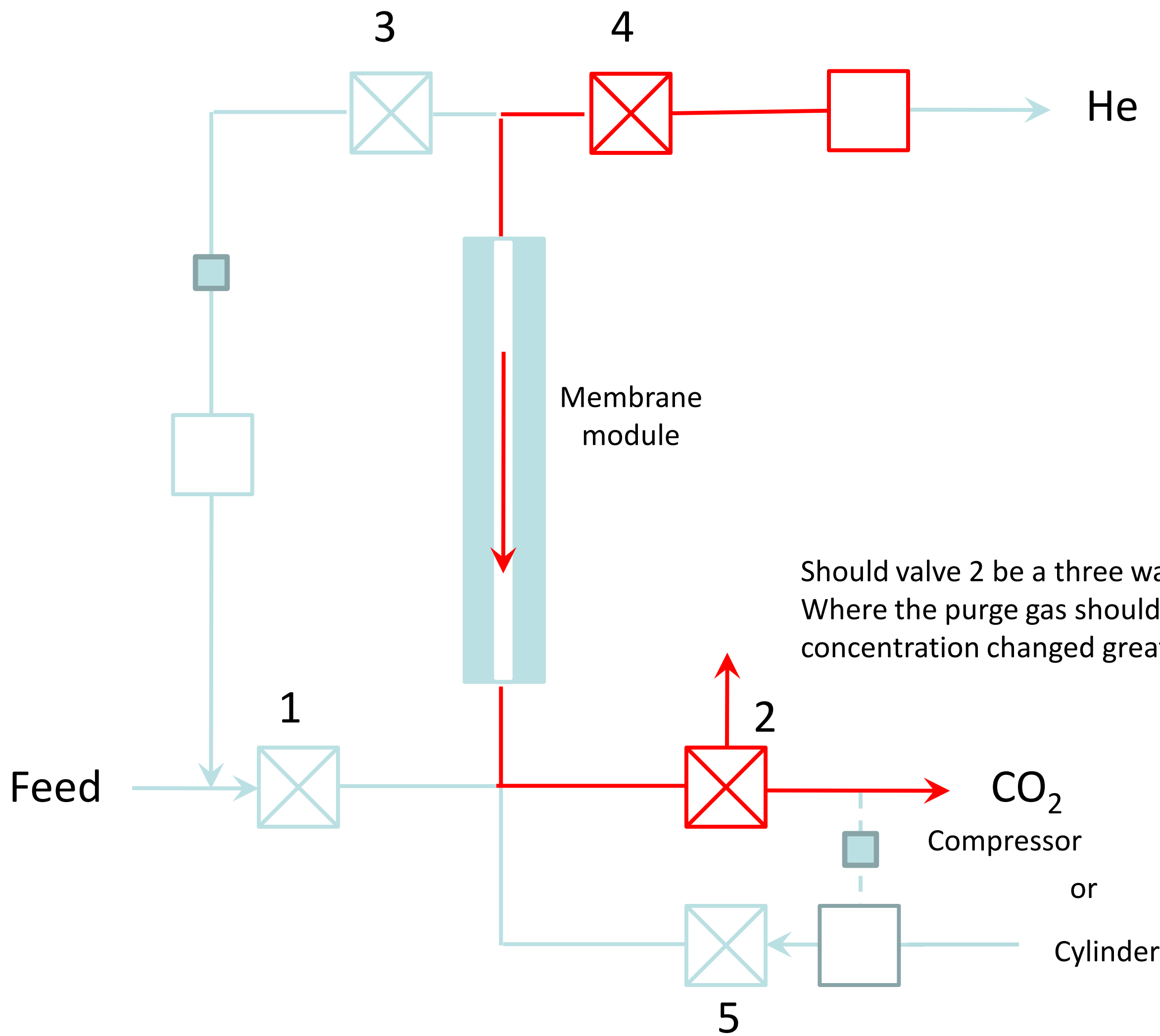
Step 2 High pressure CO_2 purge



Step 3 Countercurrent blowdown

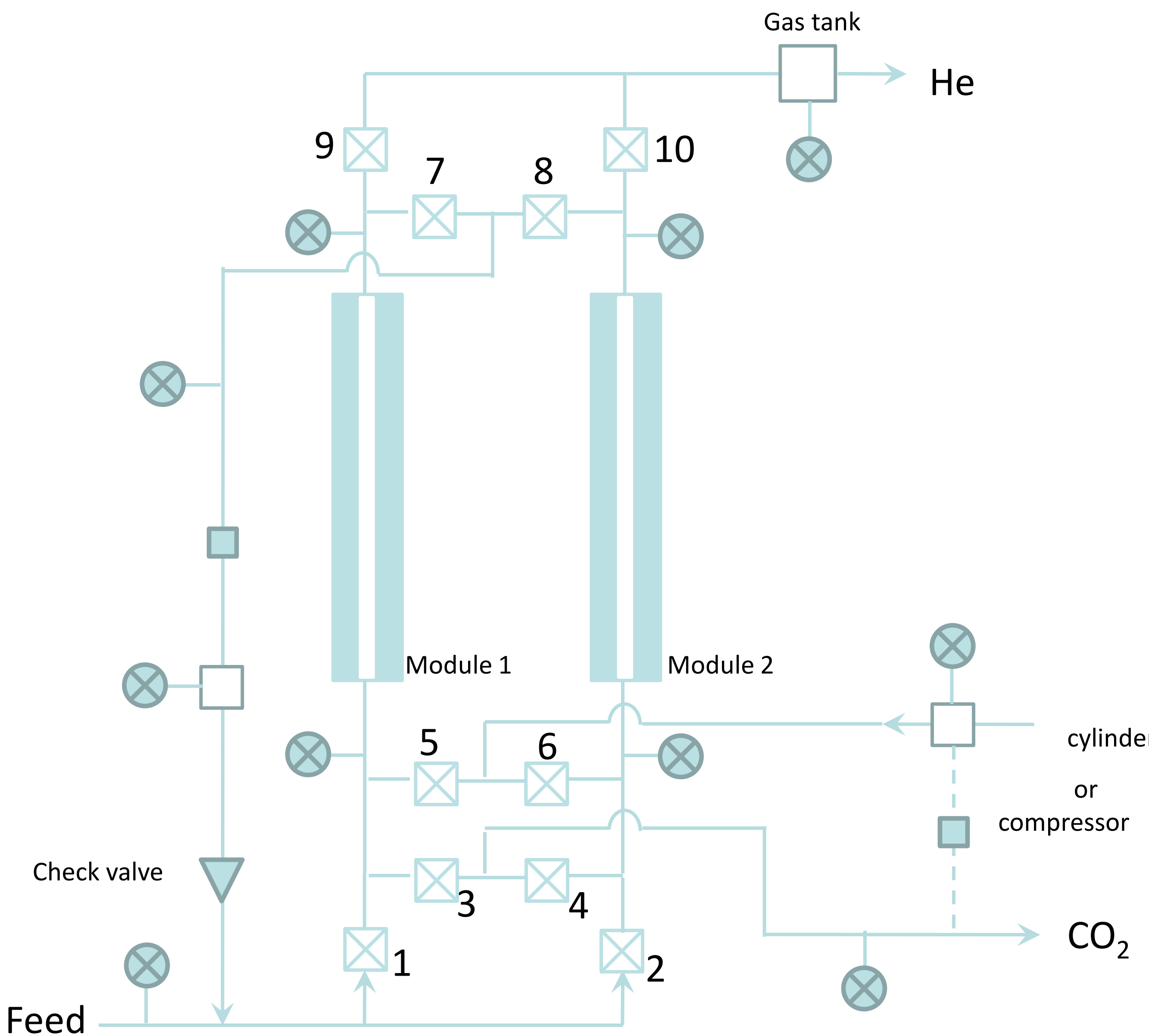


Step 4 Helium purge

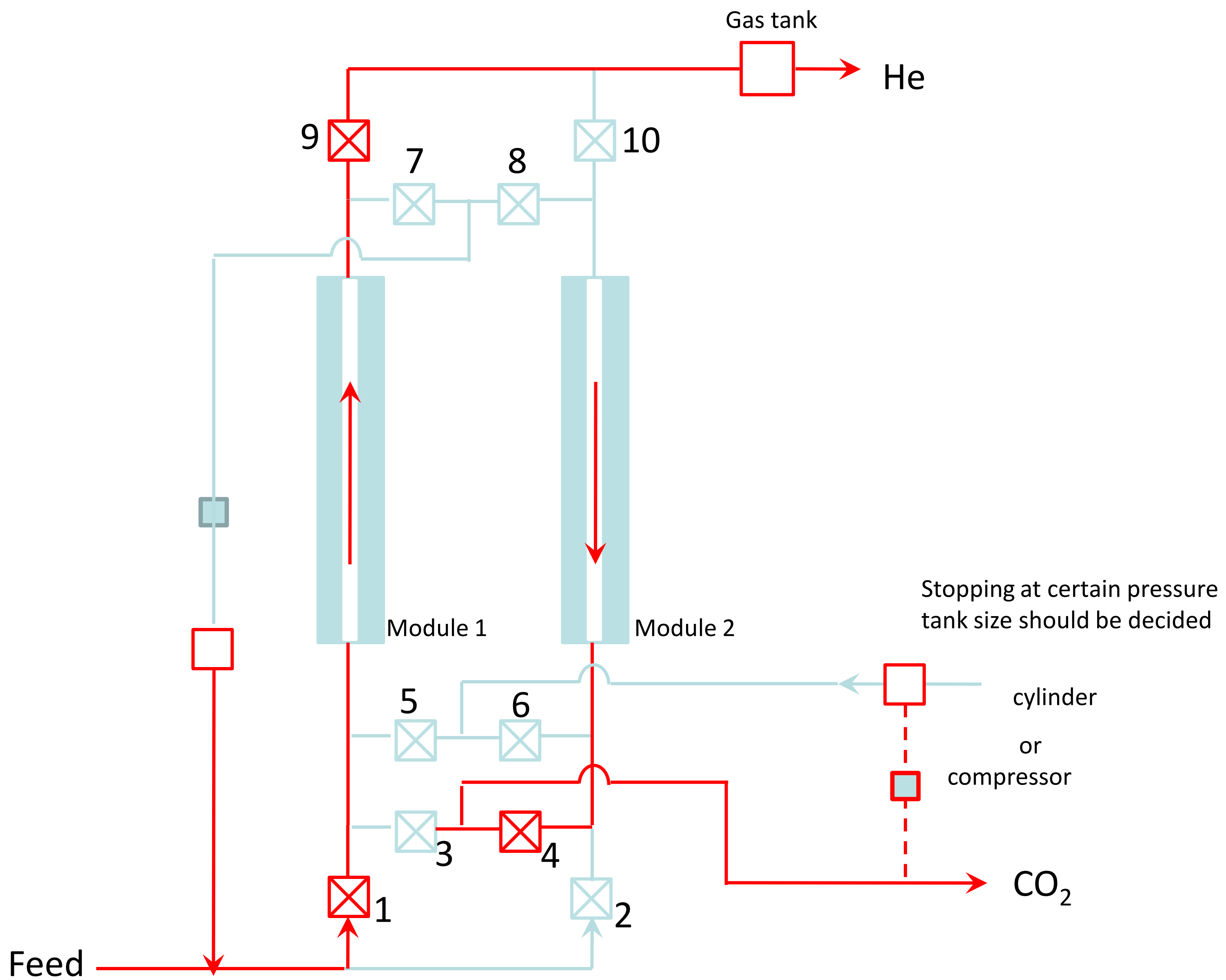


Should valve 2 be a three way solenoid valve?
Where the purge gas should go after CO_2 concentration changed greatly?

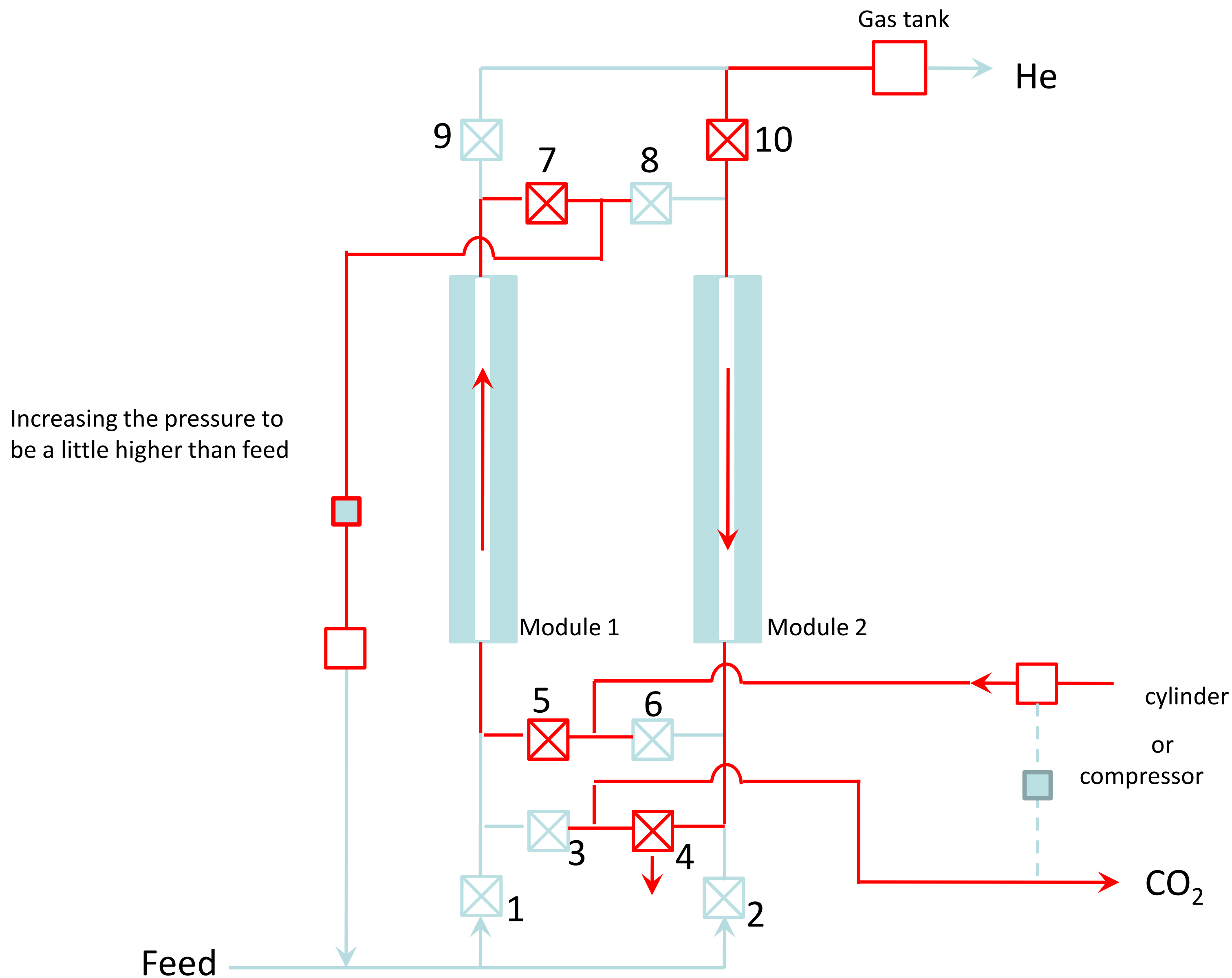
Two membrane modules cycle



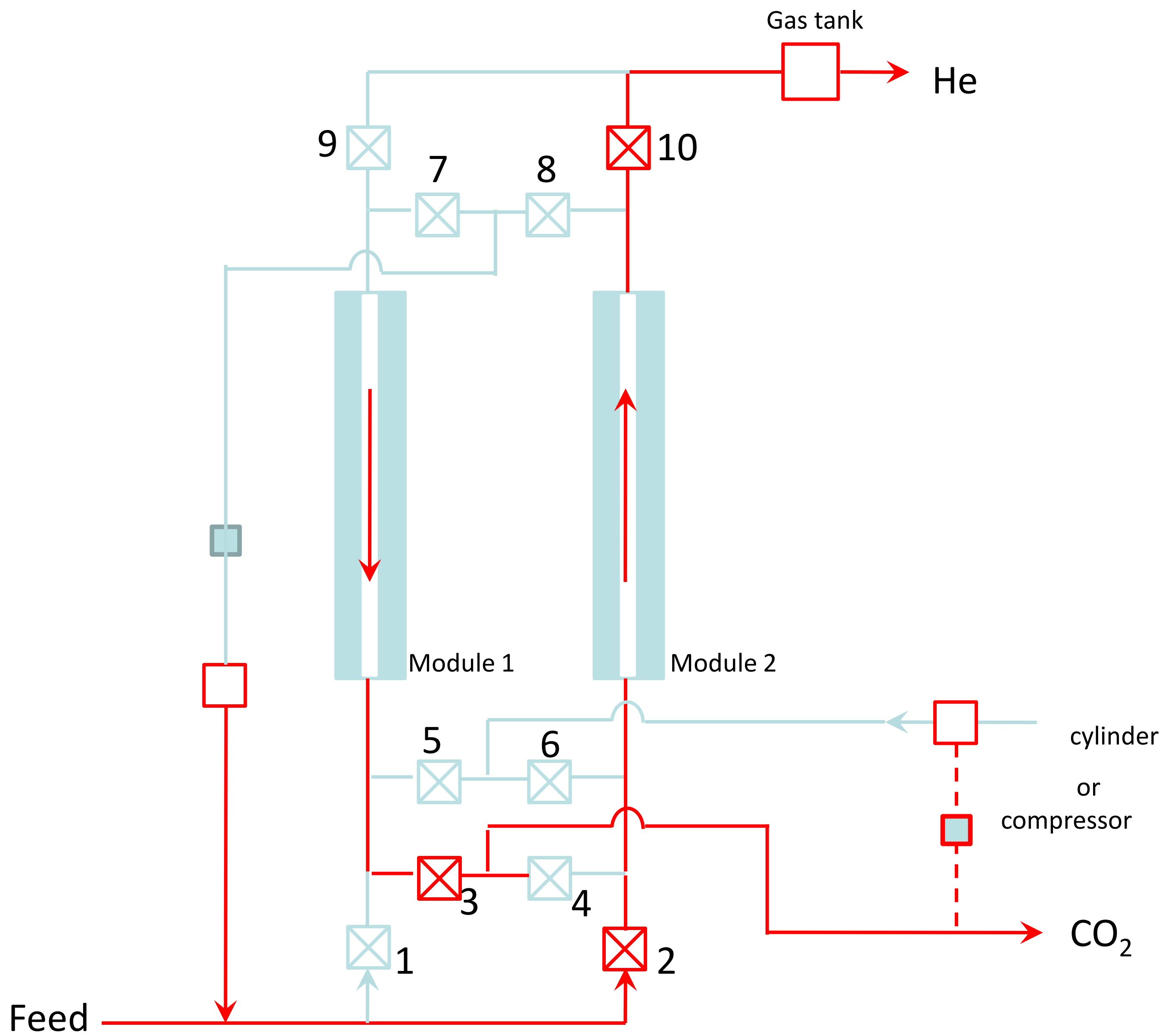
Module 1: Feed in and absorption (step 2)
Module 2: Countercurrent blowdown (step 4)



Module 1: High pressure CO_2 purge (step 3)
Module 2: Helium purge (step 1)

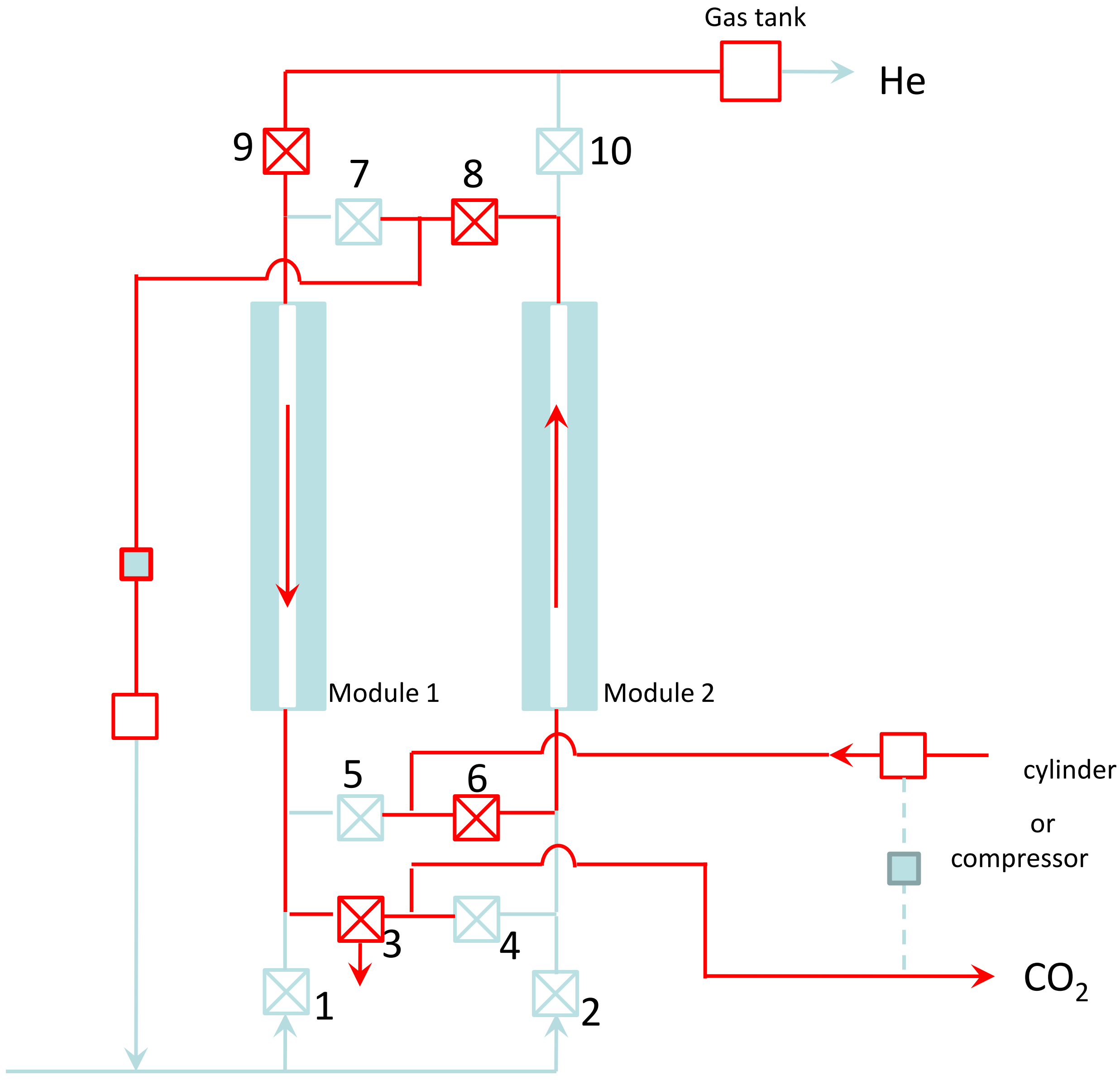


Module 1: Countercurrent blowdown (step 4)
Module 2: Feed in and absorption (step 2)



Module 1: Helium purge (step 1)

Module 2: High pressure CO_2 purge (step 3)



Tasks to be Performed: Phase III

- Task 5.0 Project Management and Planning (10/1/11 – 9/30/12)
- Subtask 5.1 Provide quarterly reports at the end of every quarter as well as the final Project Report at the end of year 3 (10/1/11 – 12/31/12)
- Task 6.0 Experimental Program and Technical Activities for Year 3 (10/1/11-9/30/12)
- Subtask 6.1 Determine the solubility and diffusivity of CO_2 and He in selected absorbents (10/1/11-6/30/12)
- Subtask 6.2 Compare mathematical model simulation results with experimental data from PSAB process (10/1/11-9/30/12)
- Subtask 6.3 Numerically explore scale up of the process to facilitate evaluation of the process (3/1/12-9/30/12)
- Subtask 6.4 Determine the loss/deterioration of the absorbents, especially amines, over extended periods (10/1/11-9/30/12)

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- Background
- Technology Description

(a) Process and Device Concept

(b) Detailed Considerations on Process and Device

- Project Objectives
- Phase I Progress
- Project Structure
- **Project Budget**
- Project Management Plan including Risk Management

Project Budget

- We have spent almost all of the money budgeted for Phase I

- Background
- Technology Description

(a) Process and Device Concept

(b) Detailed Considerations on Process and Device

- Project Objective
- Project Structure
- Project Budget
- **Project Management Plan including Risk Management**

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Project Management Plan

- Project Manager : Prof. Kamalesh K. Sirkar, PI, NJIT
- Post-Doctoral Fellow 1: Dr. Gordana Obuskovic, NJIT
- Post-Doctoral Fellow 2: Dr. Jie Xingming, NJIT
- Graduate Students: Mr. John Chau, NJIT, fully supported; Jose Sousa, limited part-time support
- Consultant : Dr. Ashok Damle, Techverse Inc., Cary, NC

The Project Manager will interact with the following companies fabricating microporous hollow fiber membranes/tubules:

1. Applied Membrane Technology, Minnetonka, MN (AMT): Stephen Conover, Thomas McEvoy, Dr. Ashok Sharma on porous hollow fiber membranes of Teflon
2. Media & Process Technology, Pittsburgh, PA (M&P): Dr. Paul K.T. Liu, Richard Ciora on coating of the surfaces of ceramic tubules of alumina
3. Porogen Inc., Woburn, MA: Dr. Ben Bikson on porous hydrophobized PEEK hollow fiber modules

Activities of PI

- **PI will work with the postdoctoral fellows (PDFs), grad. student and membrane development companies to have the membrane contactors developed**
- **PI will guide the PDFs and grad. student to design the system, order necessary equipment and supplies and have the set up built and tested**
- **PI will supervise the activities of data acquisition, device performance evaluation, estimation of properties and system modeling**
- **PI will lead the activities to take care of DOE reporting requirements and program review meetings**
- **PI will work with consultant to conduct economic evaluation of the process**
- **PI will make presentations and publications of results obtained from the project**

Risk Management

- To prevent leakage of absorbent through microporous PTFE hollow fibers having a plasma polymerized microporous fluorosilicone coating, a finer starting pore size and a provision for leakage collection at the end of tube side

- Capability of the hydrophobic coatings on ceramic tubules to hydrophobize them sufficiently (avoid defects) to eliminate leakage of absorbent into the tube side: make provision for leakage collection at the end of tube-side and a finer starting pore size

- Effect of module diameter and length on He purification ability: smallest possible tubule diameter; increase module length by connecting them in series (oven dimension limitations)

- Achieve a steady state in the cyclic process by preventing a drift in the composition and amount of two purified product streams obtained: balance cycle between absorption and regeneration; fine tune the system

Project Timeline

ID	Task Number	Task Description	Start	Finish	Task #
1	Task 1	Project Management I	10/1/2009	9/30/2010	52.14 w
2	SubTask 1.1	Status Report	10/1/2009	9/30/2010	52.14 w
3	Task 2	Experimental Program I	10/1/2009	9/30/2010	52.14 w
4	Subtask 2.1	Build Experimental Setup	10/1/2009	8/31/2010	47.86 w
5	Subtask 2.2	Develop Gas Absorption Modules	10/1/2009	9/30/2010	52.14 w
6	Subtask 2.3	Preliminary Study of PSAB	8/1/2010	9/30/2010	8.71 w
7	Task 3	Project Management II	10/1/2010	9/30/2011	52.14 w
8	Subtask 3.1	Status Report	10/1/2010	9/30/2011	52.14 w
9	Task 4	Technical Program, Year 2	10/1/2010	9/30/2011	52.14 w
10	Subtask 4.1	Study PSAB Device and Process	10/1/2010	9/30/2011	52.14 w
11	Subtask 4.2	Build Setup for Solubility and Diffusivity	10/1/2010	9/30/2011	52.14 w
12	Subtask 4.3	Develop a Model for PSAB Device and Process	10/1/2010	9/30/2011	52.14 w
13	Task 5	Project Management III	10/1/2011	9/30/2012	52.29 w
14	Subtask 5.1	Status Report	10/1/2011	9/30/2012	52.29 w
15	Task 6	Technical Program, Year 3	10/1/2011	9/30/2012	52.29 w
16	Subtask 6.1	Measure Solubility and Diffusivity	10/1/2011	6/30/2012	39.14 w
17	Subtask 6.2	Simulate Model and Compare	10/1/2011	9/30/2012	52.29 w
18	Subtask 6.3	Explore Scale up	3/1/2012	9/30/2012	30.57 w
19	Subtask 6.4	Determine Absorbent Loss	10/1/2011	9/30/2012	52.29 w

Milestone Log

PHASE I

- Milestone 1: Novel absorption module fabrication successfully completed (9/30/10)
- Milestone 2: PSAB experimental setup completed (8/31/10)
- Milestone 3: PSAB device appears to function well (9/30/10)

PHASE II

- Milestone 4: PSAB device achieving high purification of He and CO_2 streams (8/31/11)
- Milestone 5: Experimental setups for measuring solubility and diffusivity completed (9/30/11)

Milestone Log

PHASE III

- Milestone 6: Mathematical Model of PSAB developed (4/30/12)
- Milestone 7: Solubilities and diffusivities of CO_2 and He measured (4/30/12)
- Milestone 8: PSAB process simulated successfully vis-à-vis experimental performance (7/31/12)
- Milestone 9: Absorbent liquid characterized and degradation determined (9/31/12)
- Milestone 10: Scaleup and economic evaluation conducted (9/31/12)

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Closing Comments

- Special thanks to DOE Program Officer for the project, Norman Popkie
- We thank you for your attention
- I would be happy to respond to your questions